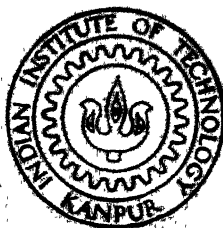


3 152-692

EFFECT OF THERMAL TREATMENT ON MICROSTRUCTURE
AND MECHANICAL PROPERTIES OF Ti - 6.5 Al - 3.3
Mo - 1.8 Zr - 0.3 Si ALLOY

by
JAIVEER SINGH YADAV

TH
ME / 1992 / 1m
yie



DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY KANPUR

JULY, 1992

EFFECT OF THERMAL TREATMENT ON MICROSTRUCTURE
AND MECHANICAL PROPERTIES OF Ti - 6.5 Al - 3.3
Mo - 1.8 Zr - 0.3 Si ALLOY

*A Thesis Submitted
In Partial Fulfilment of the Requirements
for the Degree of*
MASTER OF TECHNOLOGY

by
JAIVEER SINGH YADAV

to the
**DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY KANPUR**
JULY, 1992

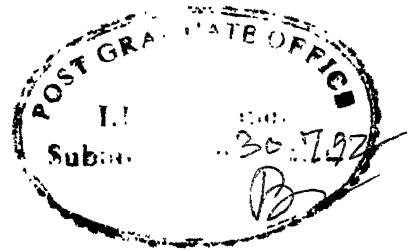
To
60-1502
yle

0 1 1992

LIBRARY

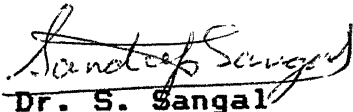
114316

ME - 1992 - M - YAD - EFF



CERTIFICATE

It is certified that the work contained in the thesis entitled, "Effect of Thermal Treatment on Microstructure and Mechanical Properties of Ti-6.5 Al-3.3 Mo-1.8 Zr - 0.3 Si Alloy" has been carried out under our supervision and that this work has not been submitted elsewhere for a degree.


Dr. S. Sangal

Assistant Professor

Department of Metallurgical Engineering
Indian Institute of Technology
Kanpur


Dr. R.C. Sharma
Professor

July, 1992

ACKNOWLEDGEMENTS

I express my heartily gratitude to Dr. R.C. Sharma and Dr. S. Sangal for their excellent guidance and constant advice throughout the course of this work. It was great pleasure working under their guidance.

Thanks are due to the Director of Aeronautics and CRE(M) for his kind sponsoring of my M.Tech. Programme. It has been a good programme of study for which I am thankful to my teachers, Dr. A.K. Jena, Dr. S.P. Gupta, Dr. M.L. Vaidya, Dr. M.N. Shetty, Dr. R.K. Dubey and Dr. S. Bhargava.

I am thankful to Mr. H.C. Srivastava, Mr. K.P. Mukherjee, Mr. V. Kumar, Mr. M.N. Mungole, Mr. B.K. Jain and Mr. Paul whose timely help at various stages of the work allowed me to complete the work. I owe my thanks to my friends Mr. Dhananjay Kumar and Mr. Nagarajan for help at various stages of work, as well as discussion on and off the subject.

My thanks are due to Mr. U.S. Misra for typing this manuscript in an elegant form at a short notice.

Last but not the least, I am sincerely indebted to my wife Mrs. Indira Yadav and daughters Km. Anjali Yadav and Richa Yadav for leaving the comfort of a settled life for my M.Tech. studies.

J.S. Yadav

CONTENTS

	Page
Acknowledgement	
List of Tables	
List of Figures	
Abstract	
 Chapter 1	
Introduction	1
 Chapter 2	
Literature Review	3
2.1 The basic development of titanium and its alloys	3
2.2 Crystal structure	4
2.3 Effect of alloying additions	4
2.3.1 Alpha stabilized systems	4
2.3.2 Beta stabilized systems	8
2.4 Classification of alloys	10
2.4.1 Alpha alloys	10
2.4.2 Near alpha alloys	12
2.4.3 Alpha-beta alloys	13
2.4.4 Near-beta alloys	13
2.4.5 Beta-alloys	14
2.5 Importance of alpha-beta alloys	14
2.6 Microstructure control in alpha-beta alloys	17
2.6.1 Microstructural features	20
2.6.2 Effect of thermomechanical working on microstructure	21
2.6.3 Effect of thermal treatment on microstructure	25
2.6.4 Effect of microstructure on mechanical properties	32
2.7 Scope of the Present Work	32
 Chapter 3	
Experimental Procedure	35
3.1 Raw material	35
3.2 Preparation of specimens	35
3.3 Thermal treatment	36
3.4 Microscopy examination	36
3.4.1 Optical microscopy	39
3.4.2 Scanning electron microscopy	39
3.4.3 Transmission electron microscopy	39
3.4.4 Quantitative microscopy	39
3.4.4.1 Volume fraction	39
3.4.4.2 Grain size	40
3.5 Mechanical properties	41
3.5.1 Hardness	41

	Page
Chapter 4	43
Experimental Results and Discussions	43
4.1 Effect of solution treatment temperature on microstructure	46
4.2 Effect of thermal treatment on microstructure	46
4.2.1 Volume fraction	46
4.2.2 Grain size	51
4.3 Effect of thermal treatment on mechanical properties	51
4.3.1 Hardness	51
4.3.2 Tensile strength	58
4.3.3 Ductility	58
4.3.4 Work hardening	65
Chapter 5	65
Conclusions and Suggestions for Further Work	65
5.1 Conclusions	65
5.2 Suggestions for further work	65

REFERENCES

LIST OF TABLES

Table	Title	Page
1	Comparison of physical properties of titanium with other metals	5
2	Beta eutectoid elements in order of increasing tendency to form compounds	11
3	Comparison of test requirements during indigen-isation and subsequent production for α - β titanium bars	18
4	Mechanical properties of selected alloys	19
5	Thermal treatment process for α - β alloys	30
6	Relative advantages of equiaxed and acicular microstructures	33
7	Effect of equiaxed vs. acicular microstructure on room temperature properties of Ti-6 Al-4V alloy	34
1	Chemical composition of experimental titanium alloy	35
1	Aging treatments after solution treatment at 950°C for 1 hour followed by air cooling	44
2	Mean volume fraction of primary alpha (\bar{V}_{α})	47
3	Mean grain size of primary alpha (\bar{x}_{α})	50
4	Work hardening parameter of solution treated and solution treated plus aged samples.	63

LIST OF FIGURES

ures	Title	Pag
. 2.1 :	Crystal structure of unalloyed titanium	6
. 2.2 :	Schematic phase diagram of alpha stabilized system	7
. 2.3 :	Schematic phase diagram of beta isomorphous system	9
. 2.4 :	Schematic phase diagram of beta eutectoid system	9
. 2.5 :	Specific tensile strength against temperature for aero engine compressor materials	15
. 2.6 :	Phase diagram that predicts results of thermomechanical working or thermal treatment practice	23
. 2.7 :	Schematic diagram of a conventional forging and subsequent heat treatment sequence for producing alpha-beta structure	24
. 2.8 :	Schematic CCT diagram showing the effect of cooling rate on the constitution of an ($\alpha + \beta$) Ti alloy	27
. 2.9 :	Schematic illustration of formation of Widmanstatten structure in Ti-6Al-4V alloy	27
. 2.10 :	Microstructural illustration of effect of β -stabilizing alloying elements on size and distribution of α -phase packets in β -processed materials (a) Ti-6-4, (b) Ti-6-62 (c) Ti-6-2-4-6	28
. 2.11 :	Schematic diagram showing microstructure together with thermal treatment temperature ranges obtained by double solution treatment and aging for ($\alpha + \beta$) alloy (by courtesy of Y. Ito)	31
. 3.1 :	Room temperature tensile specimen	37

. 4.1	: Scanning electron micrographs of samples solution treated for 1 hr and aircooled from temperatures (a) 1010 ⁰ C, (b) 980 ⁰ C, (c) 950 ⁰ C, (d) 920 ⁰ C	45
. 4.2	: Scanning electron micrographs of samples solution treated at 950 ⁰ C for 1 hr and aged at 530 ⁰ C (a) 3 hrs, (b) 6 hrs, (c) 9 hrs	48
. 4.3	: Scanning electron micrograph of sample solution treated at 950 ⁰ C for 1 hr and aged at 480 ⁰ C for 8 hrs	49
.4.4	: Scanning electron micrograph of sample solution treated at 950 ⁰ C for 1 hr and aged at 580 ⁰ C for 4.5 hrs.	49
.4.5	Thin foil micrographs of samples solution treated at 950 ⁰ C for 1 hr and aged at 530 ⁰ C for (a) unaged (b) 6 hrs (c) 12 hrs [8900X]	52
. 4.6	: Thin foil micrographs of samples solution treated at 950 ⁰ C for 1 hr & aged at 530 ⁰ C for (a) unaged (b) 6 hrs, and (c) 12 hrs [104200X]	53
. 4.7	: Effect of aging time and temperature on hardness	54
. 4.8	: Effect of aging time on tensile strength on samples solution treated plus aged at temp. 480 ⁰ C	55
. 4.9	: Effect of aging time on tensile strength on samples solution treated plus aged at temp. 530 ⁰ C	56
. 4.10	: Effect of aging time on tensile strength on samples solution treated plus aged at temp. 580 ⁰ C	57
.4.11	: Effect of aging time on ductility on samples solution treated plus aged at temp. 480 ⁰ C	59
.4.12	: Effect of aging time on ductility on samples	60

solution treated plus aged at temp. 530°C

- 4.13 : Effect of aging time on ductility on samples 61
solution treated plus aged at temp. 580°C
- 4.14 : Log-log plot of true stress-strain curve for sample 62
solution treated plus aged at 530°C for 6 hrs.

ABSTRACT

Titanium alloys have high strength to weight ratio, exceptional resistance to corrosion, creep, fatigue, fracture toughness and therefore such alloys are used in aeronautical applications. Based on the stable microstructural phases at room temperature, titanium alloys are classified as α -alloys, α - β alloys and β alloys. Alpha-beta, two phase alloys have been designed to obtain superior properties. Thermo-mechanical treatment is used to control microstructure to obtain desired mechanical properties of these two phase alloys. In this thesis, the effect of thermal treatment on microstructure and mechanical properties of Ti - 6.5 Al - 3.3 Mo - 1.8 Zr - 0.3 Si alloy has been studied. The alloy was solution treated at 950⁰C (below β transus temperature \sim 1010⁰C) and subsequently aged at temperatures varying from 480⁰C to 580⁰C for times varying from 1.5 hours to 12 hours. Volume fraction of primary α changes with varying the solution treatment temperature and does not change significantly during aging treatment. Grain size of primary α also undergoes changes with solution treatment temperature and undergoes a small change during aging. The precipitation of secondary α takes place during aging. This has been attributed as the main cause for improving the tensile strength and reducing the ductility. The hardness increases slightly with aging time as no hard phase is precipitating during aging. The aging treatment is necessary for stabilizing the microstructure for optimization of short term and long term mechanical properties.

CHAPTER 1

INTRODUCTION

Titanium alloys have high strength to weight ratio, exceptional resistance to corrosion, good stress rupture, creep, fracture toughness, fatigue strengths and therefore such alloys are used in aeronautical applications. However, most of the titanium alloys have useful strength upto 550°C . Uses of titanium alloys in aeroengine compressors has resulted in about 40% weight saving by replacing heavier heat resistant steels and super alloys used earlier [1].

Selection of any titanium alloy for specific applications usually depends on the optimisation of short and long term properties. It can be achieved by:

- . Addition of suitable alloying elements.
- . Thermo-mechanical working above and below the β -transus temperature
- . Thermal treatments

The alloying additions and treatments are used to modify the morphology of the microstructure to obtain desired mechanical properties. Based on the stable microstructural phases at room temperature, titanium alloys are classified as α -alloys, α - β alloys and β alloys.

Titanium and its alloys exhibit a broad range of phase transformations. Some of these transformations are related to the cc to hcp allotropic transformation reactions which involve the formation of metastable transition phases and/or the equilibrium phases which occur during the decomposition of metastable α or

β phases. These latter reactions usually occur in more highly alloyed situations.

The recent programme (KAVERI, LCA) of manufacturing aeroengines under Licence Production using the indigenous alloys, has been undertaken. The technical informations/data obtained from collaborators are limited. Therefore, the efforts have been made to generate more and more data and study the behaviour of indigenous alloys to improve the confidence level in aeroengine designs. In view of the above, the present work was undertaken to study the effect of thermal treatment on the evolution of microstructure and the resulting mechanical properties of Ti - 6.5 Al - 3.3 Mo - 1.8 Zr - 0.3 Si alloy. The thermal treatment includes the effect of solution treatment and the effect of subsequent aging treatment on the microstructure and mechanical properties. Mechanical testing involved the use of tensile tests and hardness tests. Microstructures were observed by optical and electron microscopy and quantified by using quantitative metallography techniques.

A broad literature review of titanium alloys and thermal treatments has been presented first, experimental procedures and results obtained have been discussed subsequently.

CHAPTER 2

LITERATURE REVIEW

2.1 The Basic Development of Titanium and its Alloys [1]

The titanium was first discovered in minerals known as rutile by W. Gregor (England) and M.H. Klaproth (Germany) in about 1790. The first commercial mill products were produced by the Titanium Metals Company of America (TMCA) around about 1950. From that time to present, production of the metal has grown at an average rate of about 8% p.a. Interest in the properties of Ti and its alloys began to accelerate during the late 1940s and early 1950s as their potential as high temperature, high-strength/weight materials with aeronautical applications became more and more widely recognized.

Although titanium is very wide spread, being the 4th most abundant structural metal occurring in the earth's crust, its lateness in achieving production status has been due to the difficulty of extracting the metal from its oxide ores [2]. Early methods of extraction and melting are still used today with more refined versions. These involve conversion of titanium oxides to titanium tetrachloride, and then, reducing with sodium or magnesium to spongy metal of high purity. Because of the reactivity of the metal in the molten state, further consolidation is done by melting under vacuum to produce metal which can be fabricated by traditional methods of forging, rolling, extrusion etc. Because of the vacuum melting techniques, titanium is essentially a very "clean" metal.

The characteristics of titanium alloys which make them as ideal materials are its high specific strength and corrosion

resistance. Its density about half that of steel, showed promise as a structural metal of moderate strength and low density which could possibly replace steel in applications where saving of weight is important.

In addition, it is about 1.6 times as dense as aluminium, with an elastic modulus about 1.5 times as high and a temperature capability well in excess of the 130°C upper limit at which aluminium alloys can withstand.

Titanium also possesses lower thermal conductivity and lower thermal expansion coefficient than aluminium and steel. Titanium is essentially non-magnetic and, because of the adherent oxide film which it carries, extremely resistant to many corrosive media, especially under oxidising conditions.

The physical properties of titanium are compared with those of other engineering metals in Table 2.1

2.2 Crystal Structure [3]

Titanium exists in two allotropic forms - alpha (a close-packed hexagonal crystal structure) and beta (a body-centered cubic structure). In pure titanium, the alpha phase is stable upto 882°C where it transforms to the beta phase. This temperature is known as the beta transus temperature. The beta phase is stable from 882°C to the melting point. These phases are illustrated in Fig. 2.1.

2.3 Effect of Alloying Additions

2.3.1 Alpha Stabilized Systems [3]

A typical binary constitution diagram for an alpha stabilized system is represented in Fig. 2.2. In this system the

Table 2.1 : Comparison of Physical Properties of Titanium with other Metals

Physical Property		Titanium	Aluminium	Copper	Iron	Magnesium
Melting Point	$^{\circ}\text{C}$	1660 \pm 10	660	1084	1535	650
Density	Mg/m^3	4.51	2.70	8.94	7.80	1.74
Thermal Conductivity at 20 $^{\circ}\text{C}$	W/m-K	17	239	385	71	147
Electrical Resistivity at 20 $^{\circ}\text{C}$	Milli Ohm m	482	26.8	17.2	97.1	44
Specific heat at 50 $^{\circ}\text{C}$	J/kg.K	528	883	389	456	1026
Coefficient of thermal expansion (0-100 $^{\circ}\text{C}$)	10^{-6}K^{-1}	8.9	24.0	16.4	11.9	25.7
Magnetic susceptibility	10^{-6}SI units	+3.43	+0.65	-0.086	Ferro-magnetic	+0.55
Elastic modulus	GN/m^2	105	70	120	200	45

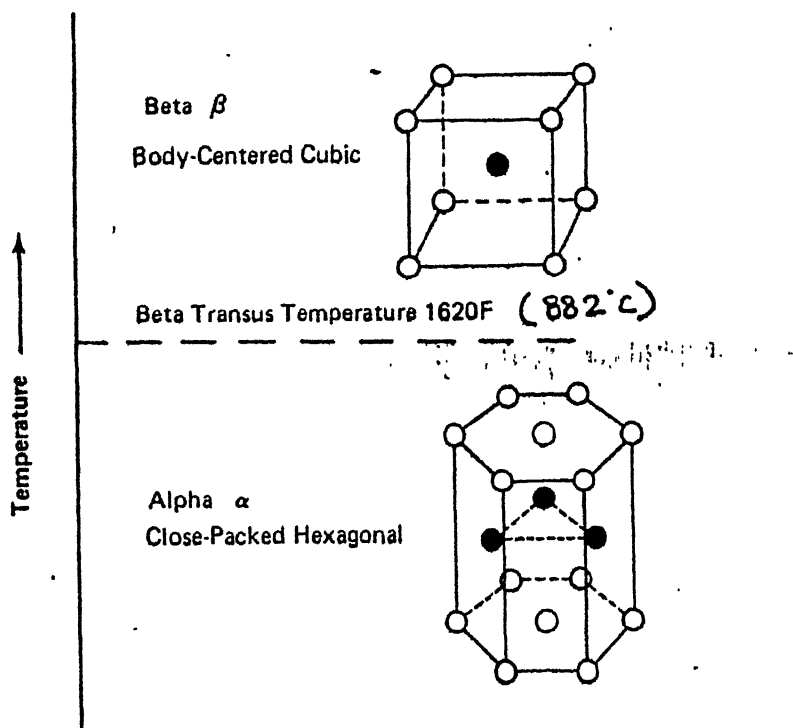


Fig. 2.1 : Crystal structure of unalloyed titanium

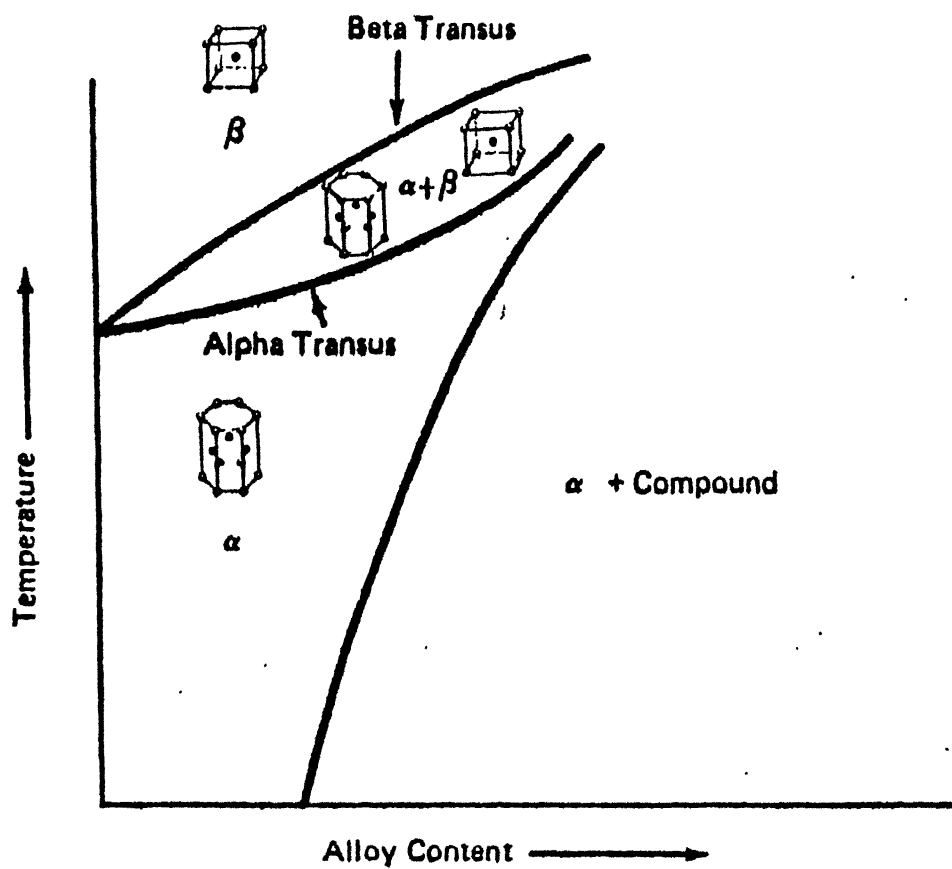


Fig. 2.2 : Schematic phase diagram of alpha stabilized system

alloying elements (Al, C, O, N) are more soluble in the α -phase. Increase in the alloy content stabilizes the α -phase to higher temperatures. That is, both the α and β transus temperatures are raised with increasing alloy content. Some of the alloying elements of the substitutional type that belong to this system are aluminium, gallium and germanium. The interstitial alloying elements of the α -stabilizing system are oxygen, nitrogen and carbon. Among all α stabilizing system, aluminium is predominant α -stabilizer. It is very effective α -strengthening element at ambient and elevated temperatures upto 550°C and is a major constituent of most commercial alloys. The low density of aluminium is an important additional advantage.

2.3.2 Beta Stabilized Systems [3]

The beta stabilized systems are classified in two groups i.e. the beta isomorphous and the beta eutectoid. Figure 2.3 shows a typical binary constitution diagram for the beta isomorphous system. In this system the alloying elements (V, Mo, Ta, Cb) are completely miscible in the beta phase and decomposition of beta to alpha plus eutectoid products does not occur even under equilibrium conditions. Increasing the alloy content decreases the alpha to beta transformation temperature. Alloying elements of the beta isomorphous type are vanadium, molybdenum, tantalum and columbium.

Figure 2.4 represents the binary phase diagram of the beta eutectoid system. In this system alloying elements (Mn, Fe, Cr, Co, Ni, Cu, Si) stabilize the beta phase, but under equilibrium conditions the beta phase decomposes to form alpha and an intermetallic compound. The beta eutectoid elements are of two

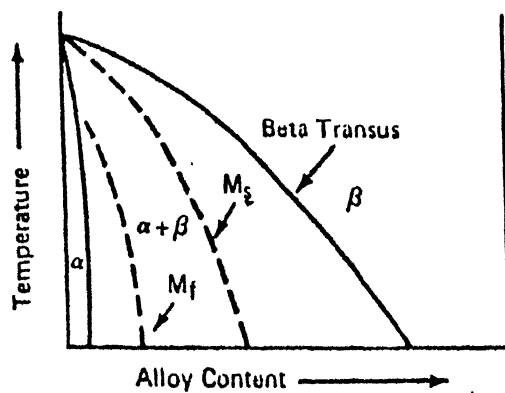


Fig. 2.3 : Schematic phase diagram of beta isomorphous system

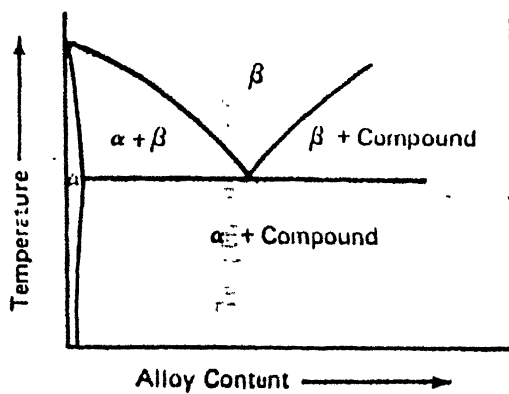


Fig. 2.4 : Schematic phase diagram of beta eutectoid system

types; the active eutectoid and the sluggish eutectoid formers. Active eutectoid formers, such as copper, nickel, cobalt and silicon, result in rapid decomposition of beta to a compound and alpha. Large amounts of elements of this type have not been used extensively in commercial alloys. The other eutectoid formers, such as chromium, iron and manganese are more sluggish in their eutectoid reactions and are not generally used in sufficient quantity to form compounds in most commercial alloys. Beta eutectoid elements arranged in order of increasing tendency to form compounds are shown in Table 2.2.

Tin and zirconium are interesting alloying elements in that they have extensive solid solubility in both the alpha and beta phases. These elements do not strongly promote phase stability, but do slow down the reaction kinetics and are useful strengthening agents. As a consequence, they are attractive alloying elements for both alpha and beta alloys.

2.4 Classification of Alloys [3]

Depending on the alloying elements added, titanium alloys can be readily classified into three groups:

- (i) alpha alloys, having hexagonal close packed structure.
- (ii) alpha-beta alloys, having a mixture of hexagonal and body centered cubic structures.
- (iii) beta alloys, having an entirely body centered structure.

2.4.1 Alpha Alloys [3]

These alloys contain only alpha stabilizers as alloying elements and hence the structure at room temperature is hcp alpha phase only. Due to the single phase nature of these alloys no

Table 2.2 : Beta Eutectoid Elements in Order of Increasing Tendency to Form Compounds

Element	Eutectoid Composition (wt%)	Eutectoid Temp. (^oC)	Composition for Beta Retention on Quenching (wt%)
Manganese	20	550	6.5
Chromium	15	675	8.0
Iron	15	600	4.0
Cobalt	9	685	7.0
Nickel	7	770	8.0
Copper	7	790	13.0
Silicon	0.9	860	--

microstructural strengthening can be achieved in these alloys. Solid solution strengthening of these alloys is also limited because an aluminium equivalent, Al^* , of more than 9.0 promotes the formation of brittle phase, α_2 (Ti_3Al). According to Rosenberg the Aluminium equivalent Al^* is expressed as below:

$$\% Al^* = \% Al + \% \frac{Sn}{3} + \% \frac{Zr}{6} + 10 (\% O + \% C + 2(\% N))$$

These titanium alloys have lower strength and limited scope for heat treatment but due to the absence of β -phase in their microstructure, have better microstructure stability and weldability. The workability of these alloys are poor due to hcp structure. Examples of these alloys are commercially pure titanium and Ti-5 Al-2.5 Sn, both of which find significant commercial applications.

2.4.2 Near Alpha Alloys [1]

These alloys fall in the group in between alpha and alpha plus beta alloys. Small additions upto 2 wt% of beta stabilizers to alpha alloys give rise to near alpha alloys. By such additions problems related to low strengths and poor hot workability of high aluminium compositions are eliminated and a compromise is effected between the higher strength which is available with alpha-beta alloys and better high temperature stability and weldability which are characteristics of alpha alloys. Examples of these alloys are Ti - 11 Sn - 2.25 Al - 5 Zr - 1 Mo - 0.25 Si, Ti-6 Al - 2 Sn - 4 Zr - 2 Mo - 0.1 Si, Ti - 6 Al - 5 Zr - 0.5 Mo - 0.3 Si all of which find applications in aeroengine components.

2.4.3 Alpha Beta Alloys [1]

The higher additions of beta stabilizers ranging from 4 to 6% in titanium alloys stabilizes the small fraction of β phase in the microstructure as illustrated in beta stabilized phase diagram [Figure 2.3]. The morphology of two phases present in such dual phase alloys can be varied by controlling various heat treatment and thermomechanical working parameters. A wide range of different property levels can therefore be achieved in these dual phase alloys. Ti - 6 Al - 4 V alloy, which is the main alloy of this group, is considered to be the "trading horse" of the titanium industry. The examples of other important alloys of this family are Ti - 4 Al - 4 Mo - 2 Sn - 0.5 Si, Ti - 6 Al - 6 V - 2 Sn, and Ti - 6.5 Al - 3.3 Mo - 1.8 Zr - 0.3 Si. This alloy has been the focus of study in the present work. All these alloys find extensive applications in the aerospace industry.

2.4.4 Near Beta Alloys [1]

Still larger additions of beta stabilizers can cause retention of beta phase at room temperature. Therefore, near beta alloys are also known as metastable beta alloys. In fact these alloys show excellent cold formability in which high strengths can also be achieved by proper thermal treatment. Examples of these alloys are Ti - 13 V - 11 Cr - 3 Al, Ti - 11.5 Mo - 6 Zr - 4.5 Sn, Ti - 10 V - 2 Fe - 3 Al. These alloys family has emerged for commercial applications only in the recent past.

2.4.5 Beta Alloys [1]

These alloys contain very large amounts of beta stabilizers which gives them high densities, poor ductilities, poor oxidation

resistance and hence little commercial value. Ti-Nb alloys come under this group. These alloys can be used as low temperature superconductors but their commercial importance is not very much. In the vocabulary of titanium industry therefore, near- β titanium alloys are referred to as beta alloys.

Since the present study is concerned with an alpha-beta type of alloy the discussion given below will be confined only to this group of titanium alloys.

2.5 Importance of Alpha-Beta Alloys

Alpha-Beta alloys have been designed to combine the better part of properties of both alpha and beta alloys. Because of the dual phase structure consisting of alpha and beta phases, a wide range of microstructural features, varying in size and morphology of both the constituent phases can be obtained in them [4] These alloys are considered as a boon for aero engine industries. In 1960 a requirement to increase the performance of the Pratt and Whitney JT3C and JT4 engines which powered early Boeing 707 and Douglas DC8 aircraft, resulted in the development of the JT3D engine. The combined results of replacing steel with α - β titanium alloys and design changes made possible in large measure by these alloys, resulted in the JT3D having 42% more take off, 13% lower fuel consumption and 18% less weight than the JT3C engine [3].

Titanium alloys show highest strength to weight ratio among all the metallic materials upto 550⁰C as illustrated in Figure [2.5]. The low density of titanium alloys pay component dividends particularly, in rotating parts. The lower the density of rotating components, less the load on these members and on those to which they are attached, thus result in weight saving and

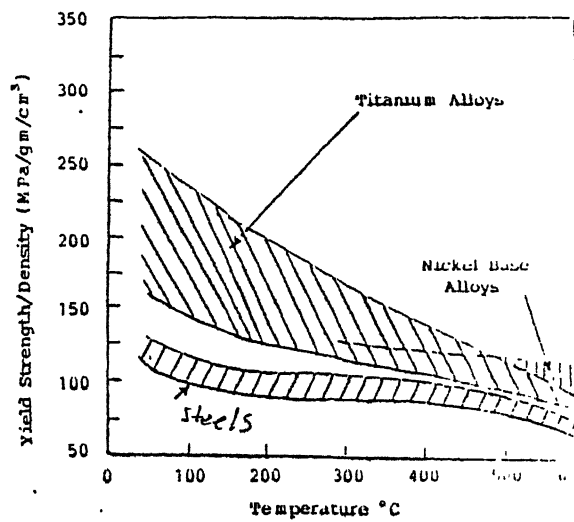


Fig.2.5 : Specific tensile strength against temperature for aero engine compressor materials

higher pay load and better fuel efficiency. The thumb rule is for \$ 1000 in fuel cost saving per pound of weight reduction. These α - β alloys are designed mainly for aeroengine compressors. The property requirements of aeroengine compressor blades and discs are summarised in brief below:

i) Blades

- With rotation speed exceeding 10,000 rpm in high pressure compressors of large engines and considerably high speed achieved in helicopter engines, compressor blades experience high centrifugal stresses. Thus specific tensile strength is the basic material requirement.
- Fatigue strength is also required to resist the additional cyclic stresses arising from forced excitation due to irregularities in the air flow distribution around the annulus caused by stators, combustion chambers, struts carrying bearings etc. and from self excitation due to blade flutter.
- Rise in exit temperature with increasing pressure ratio demands usage of materials having properties without appreciable degradation at operating temperatures.
- Resistance to erosion and impact from foreign bodies such as sand, stones and birds, is important for materials used in early stage compressor blades.
- Notch-sensitivity should be low and time for propagation of crack high.

ii) Discs

- Discs operate at high rotational speed (in the range 5000-30,000 rpm) depending on the disc size and engine type. The highest direct stresses are in the bore of the

discs where the temperature is lowest. Disc rim operates at the highest temperatures with relatively low nominal stresses. Local stress concentration associated with blade root fixing, may also cause local yielding and stress relaxation. Material requirement is -

- (a) Specific ultimate tensile strength and proof strength
 - (b) Creep strength.
- Low cycle fatigue strength is a requirement to meet the cycle stresses arising from the normal flight cycle.
 - Properties of major importance, such as crack propagation rate, fracture toughness and low cycle fatigue strength are now the design criteria.

In order to ensure that the aeroengine materials and components function reliably during their stipulated life, it is essential that the desired quality is maintained through out and property variations are within very narrow band of specifications. A typical comparison of the testing involved during indigenisation and subsequent production is shown in Table 2.3

Microstructure and grain structure influence properties significantly, where as deformation conditions and thermal treatments are the controlling factor for microstructure of a given composition alloy. Selected mechanical properties of a few titanium alloys have been shown in Table 2.4 [1].

2.6 Microstructural Control in Alpha-Beta Alloys

Microstructures are characterised by the nature of phases present, their shape, size, morphology which in turn ore functions of thermomechanical working and thermal treatment. Before we go into details lets look at some of the prominent microstructural

**Table 2.3 Comparison of Test Requirements during Indigenisation
and Subsequent Production for $\alpha - \beta$ Titanium Bars**

Sl.No.	Properties	Test requirement during	
		Indigeni- sation	Subsequent production
1.	Chemical analysis	Number of elements	Number of elements
	i) Raw materials	55	10
	ii) Product	10	10
2.	Defects (Surface + Internal)	Yes	Yes
3.	Size and tolerances	Yes	Yes
4.	Hardness	Yes	Nil
5.	Tensile*		
	RT	1	1
	HT	3	3
6.	Impact strengths (RT)	Yes	Yes
7.	Stress-rupture* (No. of tests)	8	2
8.	Creep (0.2% El)* (No. of tests)	5	Nil
9.	Fatigue* (No. of tests)	2	Nil
10.	Fracture toughness* (No. of tests)	2	Nil
11.	Metallography (Macro + Micro)	Yes	Yes
12.	Beta-transus temperature	Yes	Yes
13.	Young's modulus (No. of tests)	3	Nil
Total		99	21

* Tested at various temperatures and stress levels.

Table 2-4 Tensile Properties of Selected Alloys

Alloy	Tensile strength (min) (MPa/kg)	Yield strength(min) (MPa/kg)	Elongation (%)	Reduction in area (%)	Charpy impact strength (J)	Fatigue strength(MPa)	Fracture toughness MPa \sqrt{m}
Ti-5Al-2.5 Sn (ELI)	807	745	16	--	26	485-495	
Ti-6Al-4V	993 -1172	924 -1103	15	35	19	489-620	32-123
Ti-6Al-2Sn-4Zr-6Mo	1269	1172	10	23	--	620-751	26-34
Ti-13V-11Cr-3Al	1220	1172	8	--	--	--	--
4340 (Ultra high strength steel)	1965	1482	--	--	--	--	71
Al alloy (20240)	185.495	75-395	13-20	--	--	--	90-140
Mg alloy (A2318)	255-290	150-220	15-21	--	4	--	

Note: Range is provided in certain cases since property values depend on microstructural state. Minimum values are given where range is not specified.

features in titanium alloys.

2.6.1 Microstructural Features

- (a) Primary Alpha (α_p) - It refers to the alpha phase in a crystallographic structure that is retained from the last high temperature ($\alpha + \beta$) working or heat treatment. Morphology of primary α is influenced by prior thermomechanical history and may vary from lamellar to equiaxed grains.
- (b) Alpha Prime (α') - A supersaturated non-equilibrium hexagonal alpha phase formed by a diffusionless transformation of the beta phase. It occurs as fine randomly oriented needles.
- (c) Secondary Alpha- Alpha phase generated from α -prime on reheating in two phase field is designated as secondary alpha. It shows lamellar structure whose dimensions are function of temperature and time of heating.
- (d) Grain Boundary Alpha - Primary alpha outlining prior beta grain boundaries are referred to as grain boundary alpha. It evolves by heterogeneous transformation when cooled slowly from the beta to the alpha-beta phase field. Thickness and continuity of the layer depends on cooling rate and alloy composition.
- (e) Alpha Double Prime (α'') - It refers to a supersaturated, non-equilibrium orthorhombic phase formed by diffusionless transformation of the beta phase in certain alloys like Ti-Mo, Ti-Nb etc. which have high concentration of beta stabilizers.

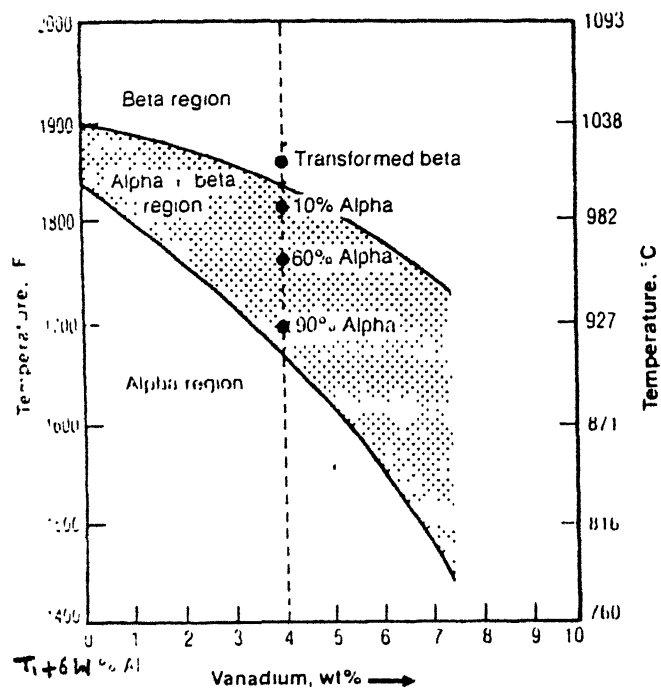
- (f) Metastable Beta - refers to the beta phase which is retained at room temperature instead of undergoing martensitic transformation. Prior beta grain boundaries are visible whose sizes are function of temperature and time of solutionizing in the beta phase field.
- (g) Alpha two (α_2) - It refers to the ordered alpha phase Ti_3Al produced by segregation of alloying elements and existing as small precipitates.
- (h) Silicides - In high silicon bearing alloys silicon combines with titanium and other alloying elements to form complex compounds known as silicides e.g. Ti_5Si_3 , Ti-Mo-Si etc. which exist as precipitates in the matrix.
- (i) Basket weave - Refers to the alpha platelets with or without interleaved beta platelets that occur in colonies in a Widemanstatten structure.
- (j) Gamma Structure - Refers to an ordered structure of Titanium - aluminium compound with a stoichiometry of Ti-Al.
- (k) Widemanstatten Structure - Refers to a structure of a platelets or alpha and beta platelets resulting from cooling from a temperature above the β transus. The platelets will often occur in colonies, giving a basket weave appearance.

2.6.2 Effect of Thermo-mechanical Working on Microstructure

Thermomechanical working of α - β alloys is governed by the deformation characteristics of the hcp alpha phase. Thermo-mechanical deformation takes place by both slip and twinning though contribution of the latter is low [5,6]. Figure

2.6 shows, schematically, the possible locations for temperature of thermomechanical working and/or thermal treatment of a typical alpha-beta alloy such as Ti - 6 Al - 4 V [13]. The higher the processing temperature in the $\alpha + \beta$ region, the more beta is available to transform on cooling. The exact form of the globular (equiaxed) alpha and the transformed beta structures produced by processing depends upon the exact location of the beta transus, which varies from heat to heat, and the degree and nature of deformation produced.

A schematic of a conventional forging and subsequent heat treatment sequence is shown in Figure 2.7. Microstructural control is basic to the successful processing of titanium alloys. Undesirable structures (grain boundary alpha, beta fleck, spaghetti or elongated alpha) can interfere with optimum property requirement. Lamellar alpha phase elongate and strain harden on working along with formation of sub-boundaries and shear bands, the misorientation of which increases with increasing degree of working [7]. Higher degree of working leads to fracturing of lamellae into small aspect ratio alpha grains [8]. Subsequent breakup to equiaxed morphology occurs during annealing when the beta phase penetrates across the lamellar width along the subboundaries or shear bands [7,9]. Nature of the starting microstructure characterised by lamellae width therefore affects the evolution of microstructures [6,10]. Other variables like grain size [7,11], strain rate [4,5,11], temperature [4,5,11], number of phases [10,11] determine amount of stored energy for static recovery and recrystallization processes which also play an important role in evolution of microstructure during annealing. Kinetics of the dynamics softening mechanisms operative which



2.6 : Phase diagram that predicts results of thermomechanical working or thermal treatment practice

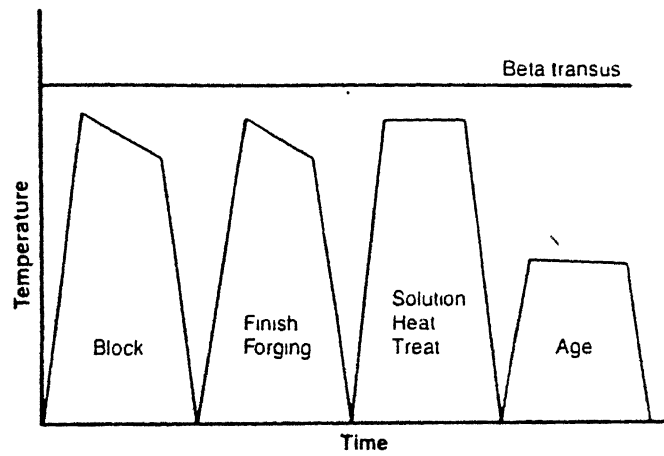


Fig. 2.7 : Schematic diagram of a conventional forging and subsequent heat treatment sequence for producing alpha-beta structure

determine the amount of stored energy is governed by these variables. Alpha-beta alloys soften by dynamic recovery and recrystallization as seen by the nature of the stress strain curve and electron microscopy observations [4,12,13].

2.6.3 Effect of Thermal Treatment on Microstructure

Thermal treatment of titanium alloys are done for a variety of reasons - to reduce residual stresses known as stress relieving, generate optimum ductility, machinability and dimensional and structural stability known as annealing, to increase strength and optimise special properties such as K_{IC} , fatigue strength, creep strength, notch fatigue, stress rupture, low cycle fatigue etc. The latter requirements are met by proper selection of thermal treatment to generate microstructures yielding requisite properties. The basis for microstructural manipulation during thermal treatment of titanium alloys centres around the beta-alpha phase transformation which takes place in these alloys during cooling. Transformation can occur either by Widmanstatten structure or martensitically depending on alloy composition and rate of cooling [14,15,16,17]. Figure 2.8 gives a schematic representation of the continuous cooling transformation diagram of an $\alpha + \beta$ alloy.

When the alloy is slowly cooled below the β transsus temperature alpha begins to nucleate at β grain boundaries with a crystallographic Burgers orientations relationship

$$\begin{aligned} (0001)_{\alpha} \quad || \quad \{ 011 \}_{\beta} \\ \langle 11\bar{2}0 \rangle_{\alpha} \quad || \quad \langle 111 \rangle_{\beta} \end{aligned}$$

Due to the close atomic matching along this common nucleation

plane, the alpha phase thickens relatively slowly perpendicular to it but grows faster along it leading to plate like structure. A schematic illustration of the formation of the Widmanstatten structure is shown in the Figure 2.9. Even when cooling at rates when nucleation and growth mechanism is operative morphology of the α phase is influenced by alloy content and cooling rates for e.g. during slow cooling (or in alloys leaner in beta stabilizer) the Widmanstatten alpha plates form in colonies or packets of plates all of which belong to the same variant of the orientation relationship between the α and β phases. With increase in cooling rates or increase in beta stabilizers content nucleation of additional variants become more prevalent and the number of plates in the Widmonstatten alpha decreases until a point is reached when the transformed region consists of a random mixture of α -plates belonging to different variants of the orientation relationship as shown in Figure 2.10.

Rapid quenching from the beta phase field leads to formation of randomly oriented needle shaped martensite (α') which is supersaturated with β stabilizers like Mo and V. Increasing β stabilizers content not only shifts the C curve for Nucleation and Growth transformation to the right leading to evolution of α' at lower cooling rates but also depresses the M_f (martensite finish temperature) the latter causing incomplete beta being found at room temperature. Subsequent aging of martensite leads to nucleation of beta phase at plate boundaries and internal substructures (such as twins) [18] followed by growth. Diffusion of excess β -stabilizing elements from supersaturated α' is utilized during growth with the latter gradually transforming to equilibrium α [19]. Resulting fine lamellar or acicular alpha

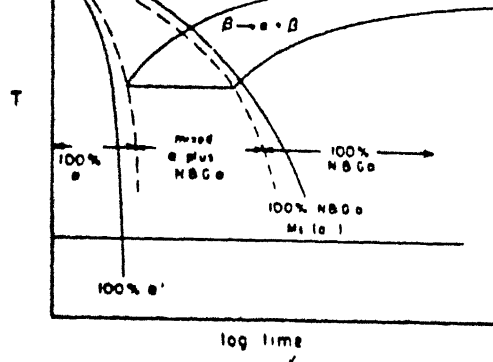
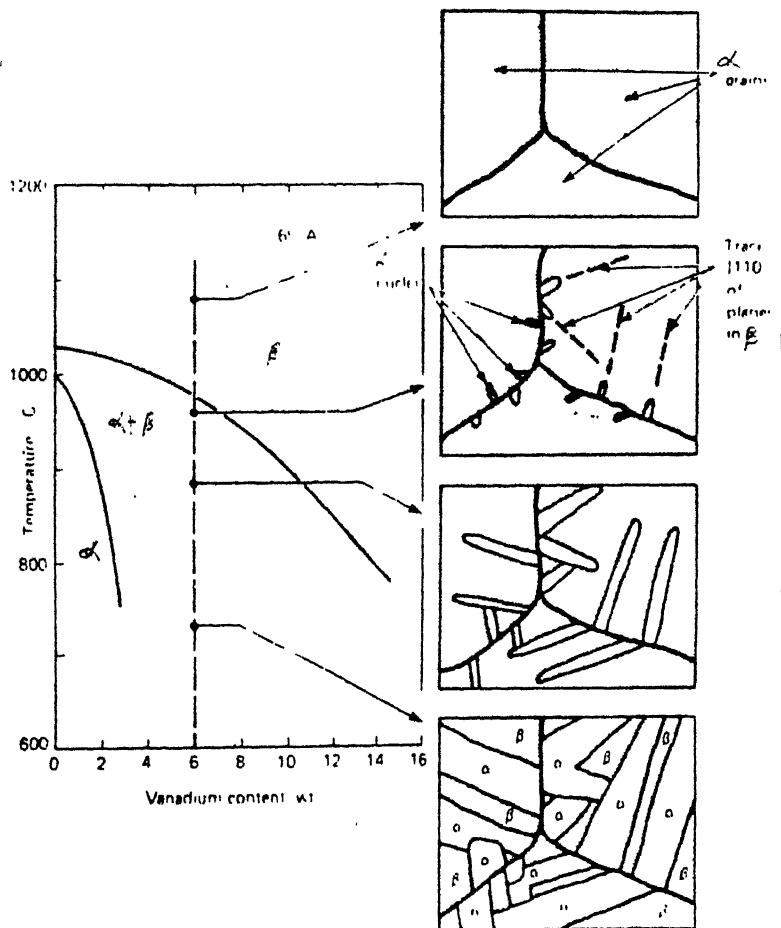


Fig. 2.8 : Schematic CCT diagram showing the effect of cooling rate on the constitution of an ($\alpha + \beta$) Ti alloy



Achieved by cooling slowly from above the β transus. Final microstructure consists of plates of α (white) separated by the β phase (dark)

Fig 2.9 : Schematic illustration of formation of Widmanstätten structure in Ti-6Al-4V alloy

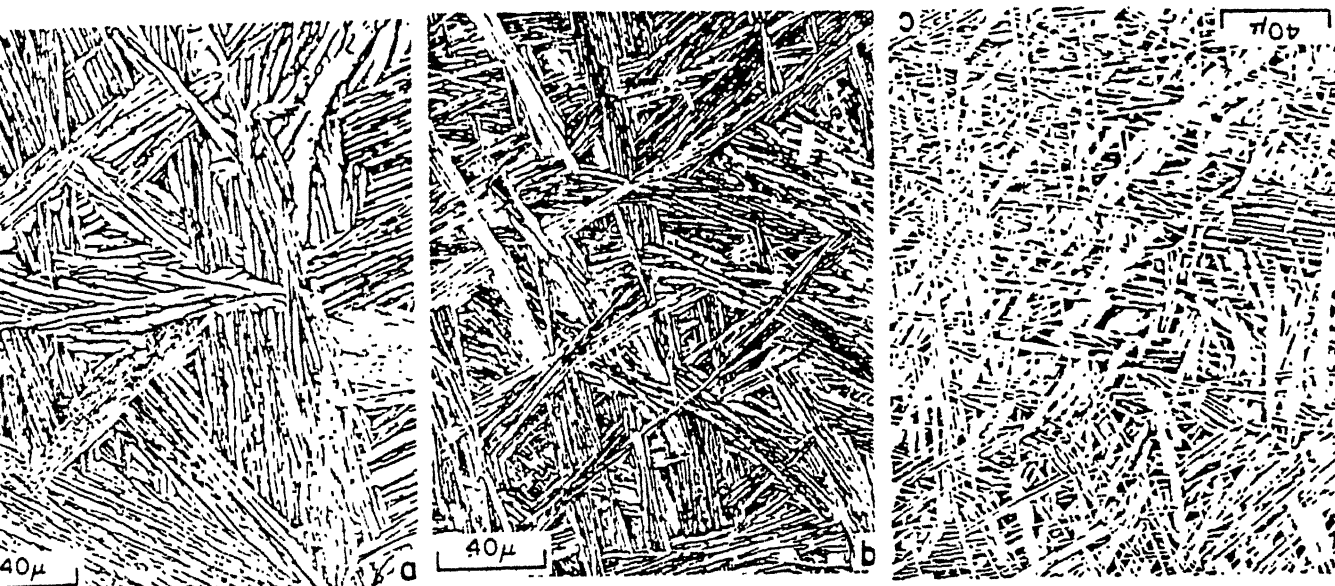


Fig. 2.10 Microstructural illustration of effect of β -stabilizing alloying elements on size and distribution of α -phase packets in β -processed materials (a) Ti-6-4 (b) Ti-6-6-2 (c) Ti-6-2-4-6

shows superior creep properties and higher fracture toughness values [1,20].

Phase transformations discussed above form the basis for deciding thermal treatment practices of α - β alloys. A common practice in the thermal treatment of these alloys consists of quenching or air cooling from a high solution treatment temperature, mainly in a range of $\alpha + \beta$, and aging subsequently at lower temperature for strengthening. Table 2.5 shows the transformations occurring during thermal treatment [21]. A solution treatment in $\alpha + \beta$ range and quench or aircooling produces equiaxed α and retained or partially transformed β phase containing hexagonal α' or orthorhombic α'' martensite. The two types [22,23] of martensite decompose to $\alpha + \beta$ or α + intermetallic compound by the subsequent low temperature aging. The retained metastable β decomposes by phase separation [24] into two bcc phase $\beta + \beta'$ of different compositions, or by forming isothermal ω phase [25] in the β phase, or by precipitating intermetallic compound such as an α_2 phase [26]. Then, the equilibrium α phase can form either from the β' or ω phase or directly [27] from the metastable β phase. One typical example [28] for the thermal treatment of $\alpha + \beta$ alloy is shown in Figure 2.11, which represents a schematic diagram showing microstructures together with heat treatment temperature ranges by double solution treatment at two separate high temperatures and aging at a low temperature for a α + β alloy. There are four different kinds of microstructures consisting of primary α , precipitated at 2nd stage and precipitated α at a lower aging temperature.

Table 2.5 : Thermal Treatment Process for $\alpha + \beta$ Alloys

Transformation	Process	Authors
Solution treatment	Quenched below β transus ($\alpha+\beta$ region) (Primary equiaxed α + retained/ partially transformed β); (Double solution treatment)	
α -phase formation	Decomposition of martensite α', α''	
	i) Hexag. $\alpha' \longrightarrow \alpha + \beta$ hexag. $\alpha' \longrightarrow \alpha + \text{compound}$	Welch [22]
	ii) Orthorhom. $\alpha'' \longrightarrow \alpha + \beta$ $\alpha'' \longrightarrow \alpha'' + \alpha' \longrightarrow \alpha + \beta$ lean rich	Ivanon [23]
	Decomposition of retained β	
	i) Isothermal $\omega_i: \beta_r \longrightarrow \omega_i + \beta$	Margolin [25]
	ii) Phase separation: $\beta_r \longrightarrow \beta' + \beta \longrightarrow \alpha + \beta$	Williams [24]
	iii) Compound precipitation $\beta_r \longrightarrow \alpha_2 + \beta$	Hamajima [26]
	iv) α -phase formation: $\beta_r \longrightarrow \alpha + \beta$	Williams and Blackburn [27]

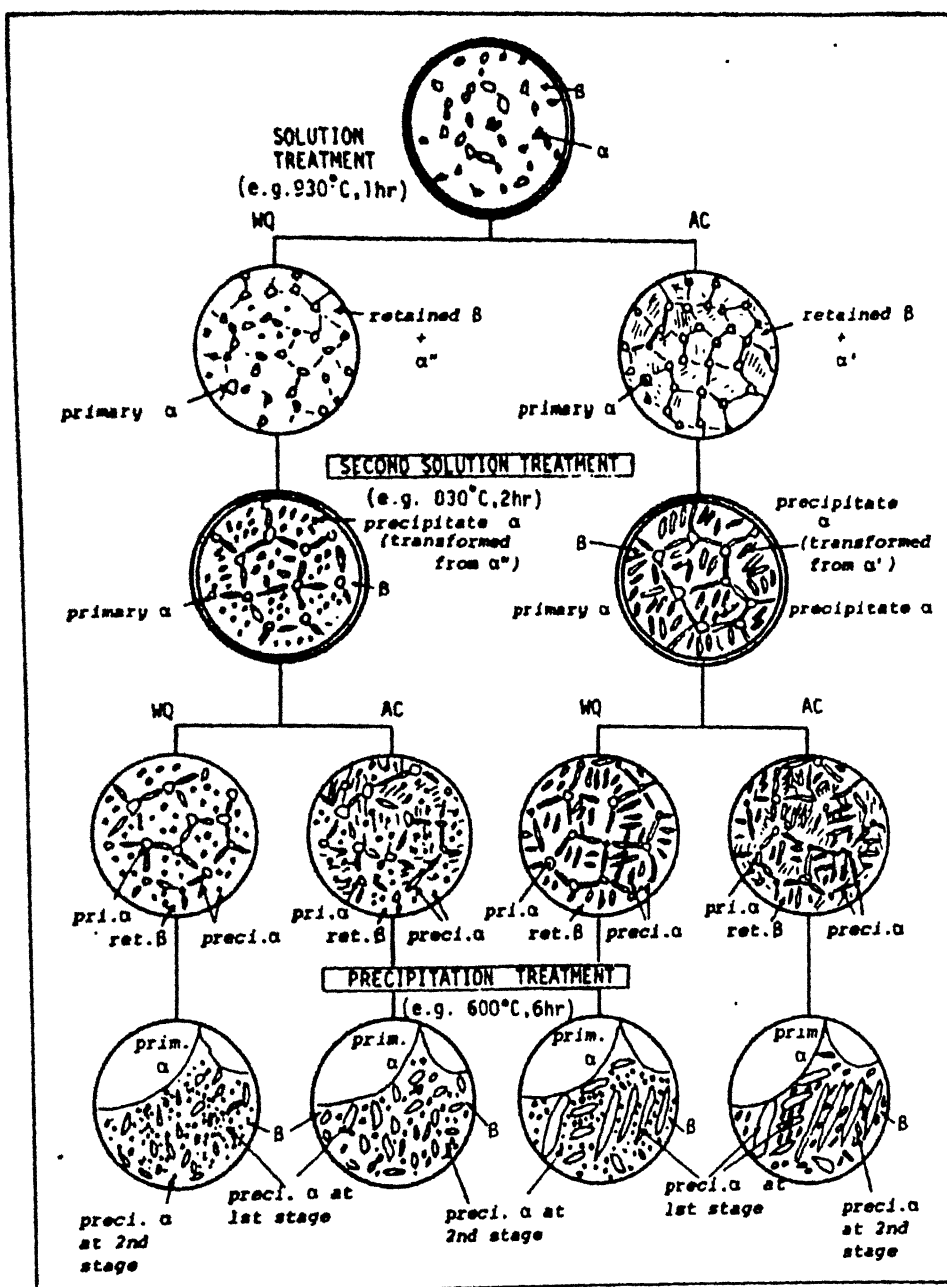


Fig. 2.11 : Schematic diagram showing microstructure together with thermal treatment temperature ranges obtained by double solution treatment and aging for $(\alpha + \beta)$ alloy (by courtesy of Y. Ito)

2.6.4 Effect of Microstructure on Mechanical Properties [1,29]

The principal microstructural variable of α -alloys is the grain size. For a fixed composition, short time strength (yield) and long time strength (creep rupture) are influenced by grain size and stored energy (if any) of deformation. For $\alpha + \beta$ alloys, the story is quite different. The α , retained β and transformed β constituents may exist in different forms, ranging from equiaxed to acicular or some combination of both. Equiaxed structures are formed by working an alloy in $\alpha - \beta$ range and annealing at lower temperatures. Acicular structures are formed by working or heat treating above the β transus and rapid cooling. Rapid cooling from temperatures high in the $\alpha - \beta$ range will result in equiaxed prior α and acicular transformed β structures.

There are property advantages and disadvantages for each type of structure. Table 2.6 compares on a relative basis, the advantages of each structure. Table 2.7 shows the actual property levels achieved by equiaxed and acicular structures produced in Ti-6Al-4V alloy.

2.7 Scope of the Present Work

The review of literature shows that although thermal treatment of Ti-6.5 Al-3.3 Mo-1.8 Zr - 0.3 Si alloy is likely to improve its properties, the detailed study has not been carried out at varying aging time and temperature. In this work, in order to investigate this aspect, effect of thermal treatment on microstructure and mechanical properties has been studied.

Table 2.6 : Relative Advantages of Equiaxed and Acicular Microstructures

Equiaxed:

- . Higher ductility and formability
- . Higher threshold stress for hot-salt stress corrosion
- . Higher strength (for equivalent heat treatment)
- . Better hydrogen tolerance
- . Better low cycle fatigue (initiation) properties

Acicular:

- . Superior creep properties
 - . Higher fracture-toughness values
-

**Table 2.7 : Effect of Equiaxed vs. Acicular Microstructure on
Room Temperature Properties of Ti - 6 Al - 4 V Alloy**

Room Temperature Property	Equiaxed	Acicular
0.2 Yield strength (MPa)	979	931
Ultimate tensile strength (MPa)	1034	1000
Elongation (%)	13	10
Fracture toughness (K_{IC} (MPa \sqrt{m})	77	93.4

CHAPTER 3

EXPERIMENTAL PROCEDURE

3.1 As Received Material

Ti-6.5 Al - 3.3 Mo - 1.8 Zr - 0.3 Si alloy (hereafter referred to as BT-9) used in this study was obtained from M/S. Mishra Dhatu Nigam Ltd. Hyderabad. The supplied material was in the form of hot rolled bars of 30 mm diameter. These bars were manufactured from an ingot which was made from titanium sponge and other master alloys by the vacuum melting of consumable electrode in the vacuum arc remelting furnace known as VAR. Double melting method for preparing the ingot was adopted to have better homogeneity and lower gases contents. Ingots thus produced were surface machined on a turning lathe and were forged, subsequently, in $\alpha + \beta$ range on 1500T hydraulic press to produce $100 \times 100 \text{ mm}^2$ billets. These billets were hot rolled to 30 mm diameter bars on a bar mill. Chemical composition of the supplied material has been shown in Table 3.1

Table 3.1 : Chemical Composition of Experimental Titanium Alloy

Alloying Elements	Al	Mo	Zr	Si	Fe	O	N	H	C	Ti
Amount (wt%)	6.47	3.33	1.75	0.26	0.20	860 ppm	150 ppm	110 ppm	90 ppm	Bal.

3.2 Preparation of Specimens

The hot rolled bars of 30 mm dia were turned to 12 mm diameter. The specimens of approximately 10 mm thickness were cut

for microscopy examinations. Tensile specimens were prepared as per the ASTM specification (E8). The dimensions of the tensile specimens are shown in Figure 3.1.

All the specimens were encapsulated in quartz tube under vacuum (0.004 Torr) to minimise the oxygen pickup during thermal treatment.

3.3 Thermal Treatment

The encapsulated specimens were thermal treated in a horizontal muffle furnace having temperature variation of $\pm 5^{\circ}\text{C}$. An additional thermocouple was kept to monitor the temperature variation. Microscopy specimens were heated for one hour at temperature ranging from 920°C to 1020°C with temperature interval of 30°C and cooled immediately in air for determination of β transus temperature. Solution treatment was carried out at 950°C , by holding the specimens for one hour and cooling in air. Aging treatment was carried out at three temperatures 480°C , 530°C and 580°C for 1.5 hours to 12 hours on solution treated specimens. These thermal treatments are summarized in Figure 3.2.

3.4 Microscopy Examination

In order to study the effect of thermal treatment on microstructure and morphology of α and β phases, specimens were prepared for microstructural examination by optical microscopy, scanning Electron microscopy (SEM) and Transmission Electron microscopy (TEM). Experimental procedures adopted for these purposes are described below:

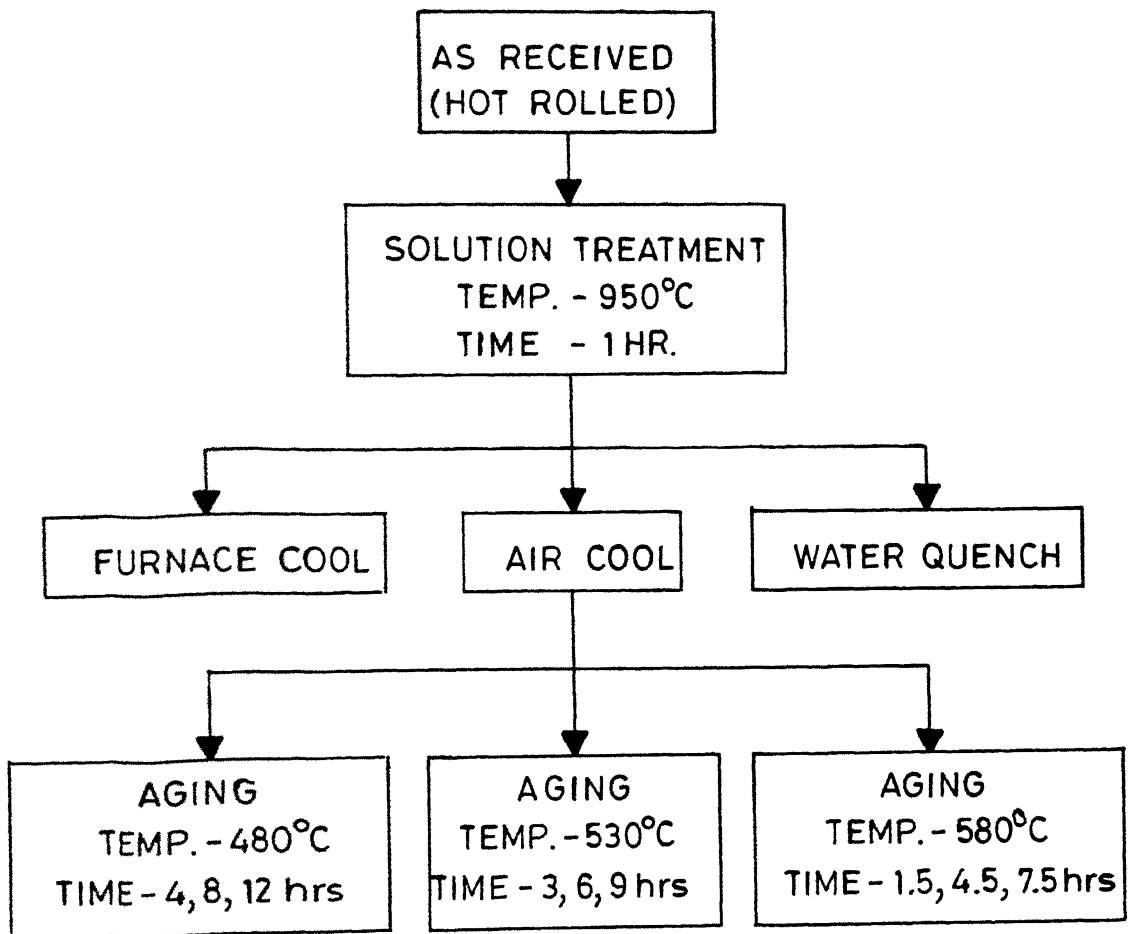


Fig. 3.2 Thermal treatment chart.

3.4.1 Optical Microscopy

Metallography specimens were cut with the observation plane transverse to the rolling direction and mechanically polished by standard methods to achieve optimum polish surface. Etching was done using Kroll's reagent (2 ml HF, 3 ml HNO_3 and 95 ml water) for 3-5 sec. Specimens were thereafter washed, dried and observed under a Lietz Metallux 3 microscope at different magnifications.

3.4.2 Scanning Electron Microscopy

Metallography specimens are mechanically polished to achieve optimum polished surface. Etching was done using Kroll's reagent (2 ml HF, 3 ml HNO_3 and 95 ml water) for 10-15 sec. Specimens were thereafter washed, dried and observed using scanning electron microscope at 15 KV.

3.4.3 Transmission Electron Microscopy

Specimens were mechanically thinned down to about 60 microns. Thereafter discs of 3 mm diameter were punched out. These discs were electrojet polished in a double jet polisher using standard electropolishing techniques. The electrolyte used consisted of 90 ml perchloric acid, 52 ml butanol and 900 ml methanol, at the temperature of -40°C and voltage 30 V. Jet polished specimens were observed in the EM 430T transmission electron microscope (Philips make) at 300 kV.

3.4.4 Quantitative Microscopy

3.4.4.1 Volume Fraction

For determining the volume fraction of primary α and transformed β phases, the point counting method (ASTM

specification E 562) has been adopted [30]. A grid consisting of 49 points systematically placed, has been applied on a microphotographs (1000X) at random and the number of points lying in the phase of primary α (P_α) are counted. Points lying on the phase boundary are counted as one-half. The point fraction which is the measure of volume fraction is defined as

$$V_\alpha = P_p = \frac{\sum P_\alpha}{n P_0}$$

where n = number of fields

P_0 = number of grid points (49 in this case)

and the confidence interval is given by [31]

$$V_\alpha \pm Z \sigma$$

where, σ , the standard deviation of the mean is given by

$$\sigma^2 = \frac{V_\alpha (1 - V_\alpha)}{\text{Total number of grid points}}$$

Using the above expressions, 90% confidence intervals were determined by taking the value of Z to be 1.64. The main assumption made in the above analysis is that the mean values are normally distributed.

3.4.4.2 Grain Size

For measuring the grain size of primary α phase, average intercept length method (ASTM specification E112) has been adopted [30]. The average intercept length \bar{L}_α which is the measure of grain size is defined as,

$$\bar{x} = \bar{L}_\alpha = \frac{L_T \times V_\alpha}{N \times M}$$

where L_T = total length of test line

N = total number of particles/grains intercepting the test line

M = magnification

V_v = volume fraction

Total of 8 fields were measured on each sample. Because of the rather small number of fields measured, 90% confidence interval was measured by assuming the mean value to be distribution on t-distribution. Thus, the 90% confidence interval is given by [31]

$$\bar{X} \pm t \sigma$$

where

$$\sigma = \frac{S}{\sqrt{n}}$$

and

$$S = \sqrt{\frac{\sum (X - \bar{X})^2}{n-1}}$$

X = grain size

\bar{X} = mean grain size

n = number of observations

t = 1.895 for 90% confidence (for n=8).

3.5 Mechanical Properties

3.5.1 Hardness

Vickers hardness measurement was done on thermal treated specimens on a Vickers hardness tester model No. 6948 (Matsuzawa Seiki Co. Ltd. Japan) using the indentation load of 30 kg. Three indentations were taken on each polished specimen and the average value was reported.

3.5.2 Tensile Strength

Tensile testing at room temperature was done on thermal treated specimens to determine the 0.2% yield strength, ultimate tensile strength, percentage elongation on 5d gauge length and percentage reduction in area, on Instron 1196, as per the ASTM specification E8, keeping the full scale load 50 KN, chart speed 20 mm/min and cross head speed 0.5 mm/min.

CHAPTER 4

EXPERIMENTAL RESULTS AND DISCUSSIONS

As mentioned in Chapter 3, volume fraction of primary α reduces with increasing the solution treatment temperature and two phase ($\alpha + \beta$) structure changes to single phase at and above β transus temperature. In order to study the effect of thermal treatment on microstructures and mechanical properties of Ti-6.5 Al-3.3 Mo-1.8 Zr - 0.3 Si alloy (referred as BT-9), the β transus temperature has been determined initially and then samples were solution treated at 950°C for 1 hr and aged at three different temperatures i.e. 480°C, 530°C and 580°C (see Table 4.1). Results obtained by these treatments have been presented and discussed in this chapter.

4.1 Effect of Solution Treatment Temperature on Microstructure

Solution treatment temperature affects both the volume fraction and grain size of $\alpha + \beta$ structure. As the solution treatment temperature increases the volume fraction of primary α reduces and becomes zero at β transus temperature. Figure 4.1 shows the microstructural changes in the samples solution treated at temperature 920°C, 950°C, 980°C and 1010°C. By observing the microstructures, it has been found that the β transus temperature for the alloy is ~1010°C at which two phase ($\alpha + \beta$) structure changes to single phase (acicular α known as martensite) after air cooling. This temperature is in agreement with data in literature [1] for this alloy. The best combination of mechanical properties is obtained when the structure contains about 20-30

**Table 4.1 : Aging Treatments after Solution Treatment at 950°C
for 1 hour followed by Air Cooling**

Aging temperature (°C)	Aging (time) (hours)
480°C	4
	8
	12
530°C	3
	6
	9
580°C	1.5
	4.5
	7.5



a



b



c



d

Fig. 4.1 : Scanning electron micrographs of samples solution treated for 1 hr and aircooled from temperatures (a) 1010°C , (b) 980°C , (c) 950°C , (d) 920°C

percent equiaxed alpha [1]. Therefore, the solution treatment has been carried out at 950°C for 1 hr and air cooled, which provides the 28% equiaxed alpha (see table 4.2).

4.2 Effect of Thermal Treatment on Microstructure

4.2.1 Volume Fraction

Volume fraction of primary α has been measured on the solution treated and solution treated plus aged samples by the point count technique discussed in section 3.4.4.1. The results of the volume fraction measurements of primary α is listed in Table 4.2. The table shows both the mean values and the 90% confidence interval. From these values it may be concluded that there is no significant change in the volume fraction of primary alpha during aging treatments. Measurements of the size and volume fraction of the secondary alpha phase which precipitates during aging from metastable β were difficult because of its fine size. Therefore these measurements could not be made.

4.2.2 Grain Size

Figure 4.2 shows the microstructure of samples solution treated at 950°C for 1 hr and aged at 530°C for 3 hrs, 6 hrs and 9 hrs. Figures 4.3 and 4.4 show the microstructures of samples solution treated at 950°C for 1 hr and aged at 480°C for 8 hrs and 580°C for 4.5 hrs. Mean grain size of primary α has been measured by the intercept length method in the solution treated and solution treated plus aged specimens. Table 4.3 shows both the mean grain size and the 90% confidence interval. In all the samples the shape of primary alpha has been found equiaxed. The

Table 4.2 : Mean Volume Fraction of Primary α (\bar{V}_α)

Thermal Treatment	Mean Volume Fraction (\bar{V}_α)	Range for 90% confidence $\bar{V}_\alpha \pm 1.64\sigma_{\bar{V}_\alpha}$
950 ⁰ C/1h/AC	0.28	0.28 \pm 0.05
950 ⁰ C/1h/AC+480 ⁰ C/4h/AC	0.30	0.30 \pm 0.05
950 ⁰ C/1h/AC+480 ⁰ C/8h/AC	0.31	0.31 \pm 0.05
950 ⁰ C/1h/AC+480 ⁰ C/12h/AC	0.31	0.31 \pm 0.05
950 ⁰ C/1h/AC+530 ⁰ C/3h/AC	0.30	0.30 \pm 0.05
950 ⁰ C/1h/AC+530 ⁰ C/6h/AC	0.31	0.31 \pm 0.05
950 ⁰ C/1h/AC+530 ⁰ C/9h/AC	0.32	0.32 \pm 0.05
950 ⁰ C/1h/AC+580 ⁰ C/1.5h/AC	0.31	0.31 \pm 0.05
950 ⁰ C/1h/AC+580 ⁰ C/4.5h/AC	0.32	0.32 \pm 0.05
950 ⁰ C/1h/AC+580 ⁰ C/7.5h/AC	0.33	0.33 \pm 0.05



a



b



c

Fig. 4.2 : Scanning electron micrographs of samples solution treated at 950°C for 1 hr and aged at 530°C (a) 3 hrs, (b) 6 hrs, (c) 9 hrs



Fig.4.3 : Scanning electron micrograph of sample solution treated at 950°C for 1 hr and aged at 480°C for 8 hrs

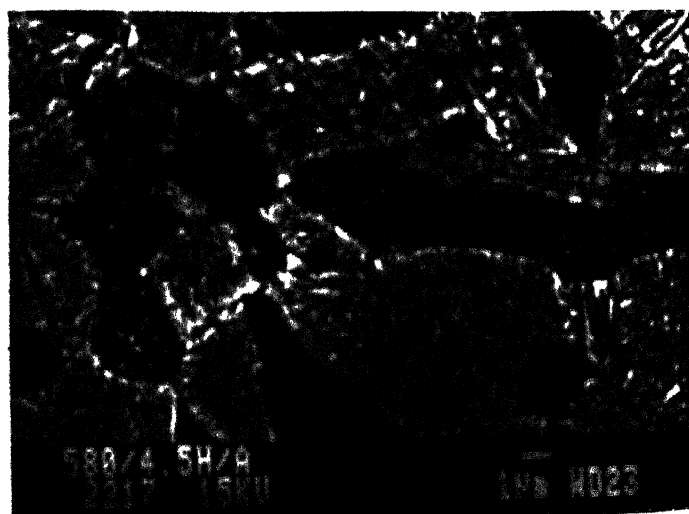


Fig.4.4 : Scanning electron micrograph of sample solution treated at 950°C for 1 hr and aged at 580°C for 4.5 hrs.

Table 4.3 : Mean Grain Size of Primary α (\bar{x}_α)

Thermal Treatment	Mean Grain size \bar{x}_α (μm)	Range for 90% Confidence $\bar{x}_\alpha \pm t \sigma$
950 ^o C/1h/AC	1.66	1.66 \pm 0.14
950 ^o C/1h/AC+480 ^o C/4h/AC	1.84	1.84 \pm 0.14
950 ^o C/1h/AC+480 ^o C/8h/AC	1.95	1.95 \pm 0.14
950 ^o C/1h/AC+480 ^o C/12h/AC	2.01	2.01 \pm 0.12
950 ^o C/1h/AC+530 ^o C/3h/AC	1.95	1.95 \pm 0.15
950 ^o C/1h/AC+530 ^o C/6h/AC	1.96	1.96 \pm 0.14
950 ^o C/1h/AC+530 ^o C/9h/AC	1.99	1.99 \pm 0.14
950 ^o C/1h/AC+580 ^o C/1.5h/AC	1.92	1.92 \pm 0.17
950 ^o C/1h/AC+580 ^o C/4.5h/AC	2.06	2.06 \pm 0.17
950 ^o C/1h/AC+580 ^o C/7.5h/AC	2.11	2.11 \pm 0.17

example, aging at 580°C for 7.5 hours results in a significant increase in grain size from 1.66 μm to 2.11 μm . The metastable β which decomposes to stable beta and secondary α [2] during aging. This is clearly confirmed in the TEM micrographs (Figures 4.5 and 4.6). The secondary alpha lamelle thickness increases with increasing aging time.

4.3 Effect of Thermal Treatment on Mechanical Properties

4.3.1 Hardness

Vickers hardness measurement has been done for solution treated sample and solution treated plus aged specimens. The average Vickers hardness values of three indentions have been plotted against the aging time in Figure 4.7. There is no significant increase in hardness values as can be seen from Figure 4.7. Scanning micrographs and TEM observations also confirm that there is no precipitation of α_2 phase taking place during aging. The metastable beta which transform to stable beta and secondary alpha, does not increase significantly in hardness values that there is no precipitation of hard phase during aging which is also justified by observing the microstructures in section 4.2. The metastable β phase which transformed to stable β and secondary alpha during aging [30] does not increases the hardness.

4.3.2 Tensile Strength

Figures 4.8, 4.9 and 4.10 show that the tensile strength increases with increase in aging time. The yield strength of material is related to grain size through the Hall-Petch relationship

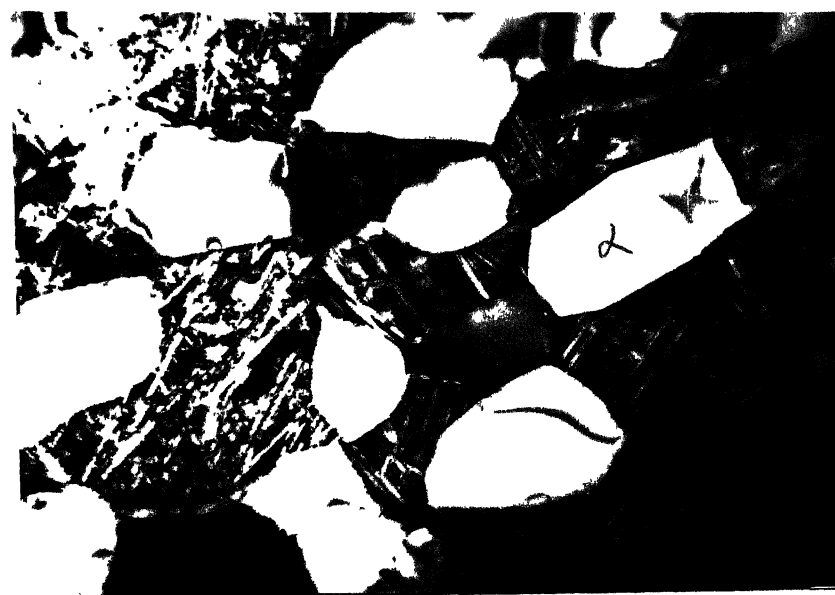
$$\sigma = \sigma_0 + K d^{-1/2}$$



a

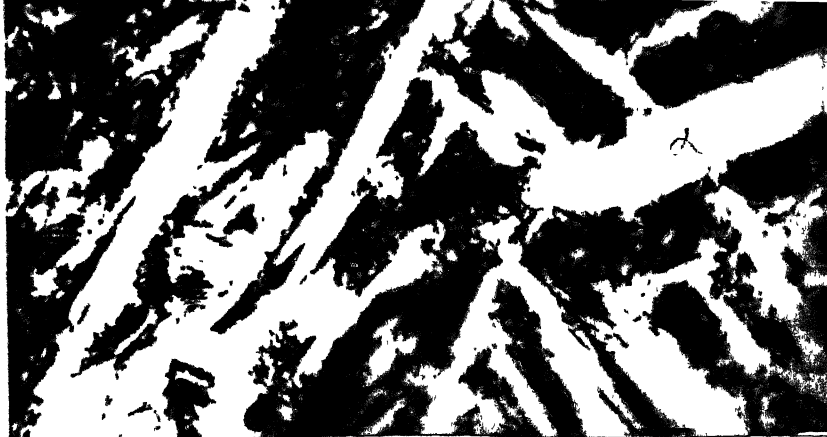


b



c

Fig.4.5: Thin foil micrographs of samples treated at 950°C for 1 hr and aged at 530°C solution for (a) unaged (b) 6 hrs (c) 12 hrs [8900X]



a



b



c

Fig. 4.6 : Thin foil micrographs of samples solution treated at 950°C for 1 hr & aged at 530°C for (a) unaged (b) 6 hrs, and (c) 12 hrs [104200X]

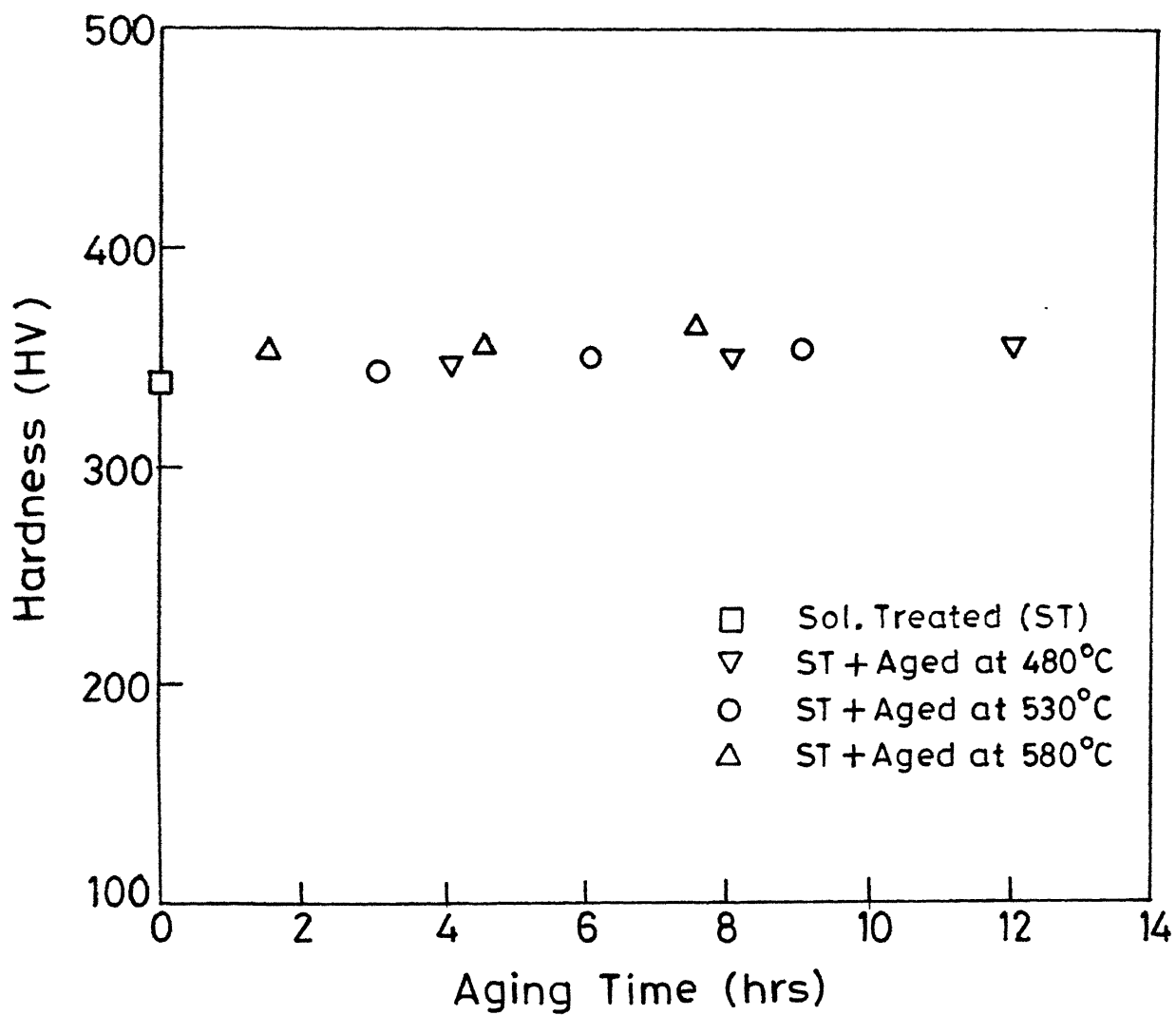


Fig.4.7 Effect of aging time and temperature on hardness.

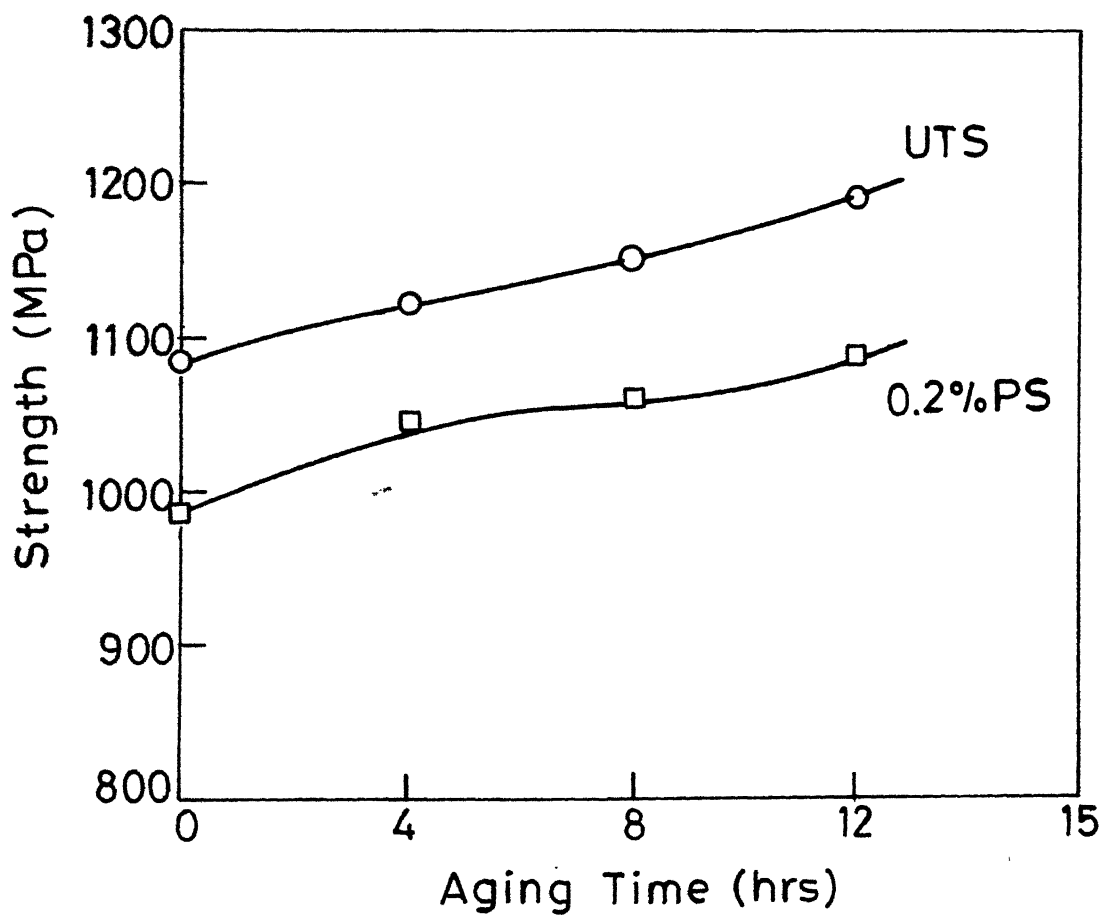


Fig.4.8 Effect of aging time on strength at temperature 480°C

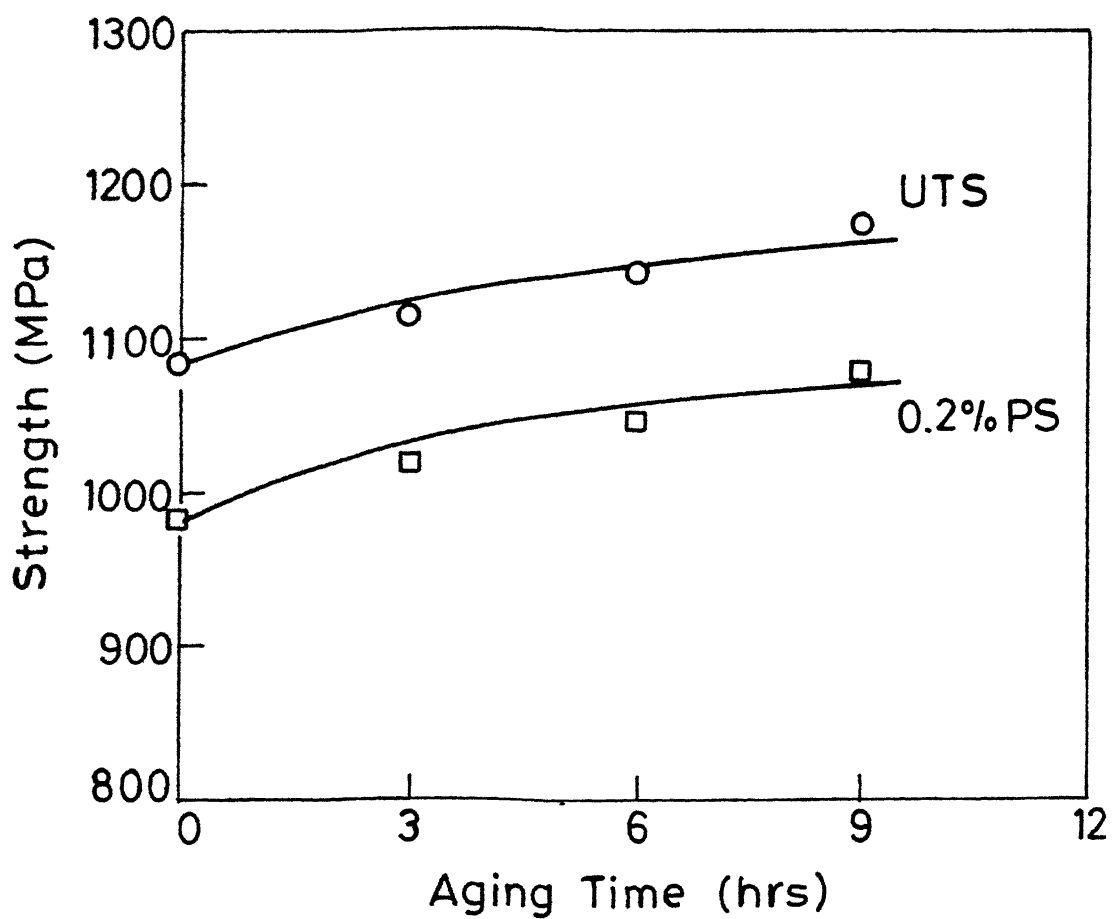


Fig. 4.9 Effect of aging time on strength at temperature 530°C.

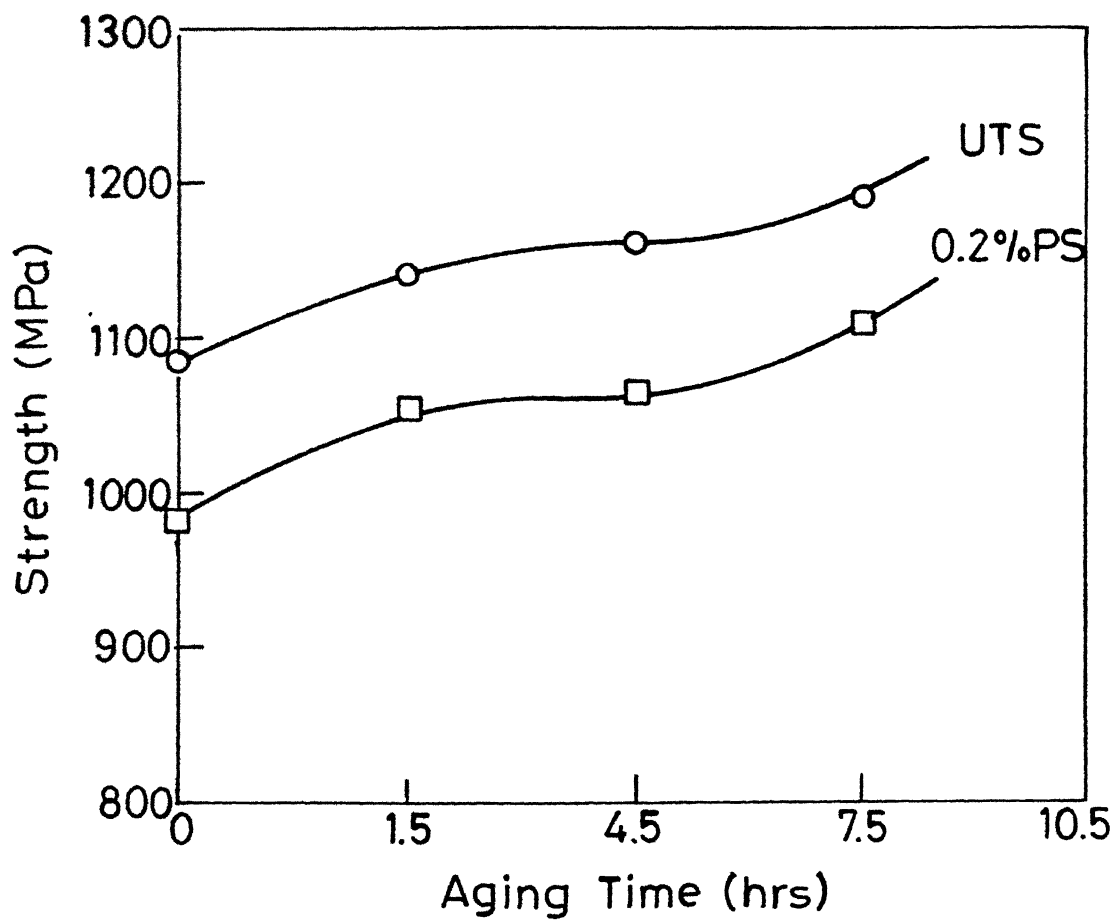


Fig.4.10 Effect of aging time on strength at temperature 580°C.

where d , the effective grain size, is defined by the mean free path for slip in the structure. Solution treatment below β transus temperature ($\sim 1010^{\circ}\text{C}$) breaks up the prior β grain structure. The metastable beta transforms to stable beta and secondary alpha during aging. This precipitation of secondary alpha reduces the mean free path for slip and therefore results in increase in the tensile strength.

4.3.3 Ductility [22,23]

Figure 4.11, 4.12 and 4.13 show the effect of aging time on the ductility. It has been observed that % elongation and reduction in area decrease with increase in aging time. As discussed in section 2.6.3, the metastable beta phase transforms to stable beta and secondary alpha during aging. The ductility of hcp (α) phase is poor compared to the bcc (β) phase. The mean free path for slip decreases during aging of solution treated samples and therefore cross slip becomes more difficult. The piling up of dislocations increases the void formation and crack propagation rate which results in reduction in the ductility of solution treated plus aged samples.

4.3.4 Work Hardening [33]

The work hardening parameter (n) is expressed as

$$\sigma = K \epsilon^n$$

where σ = true stress

ϵ = true strain

K = strength coefficient

The slope of the $\log \sigma$ vs. $\log \epsilon$ plot gives the work hardening

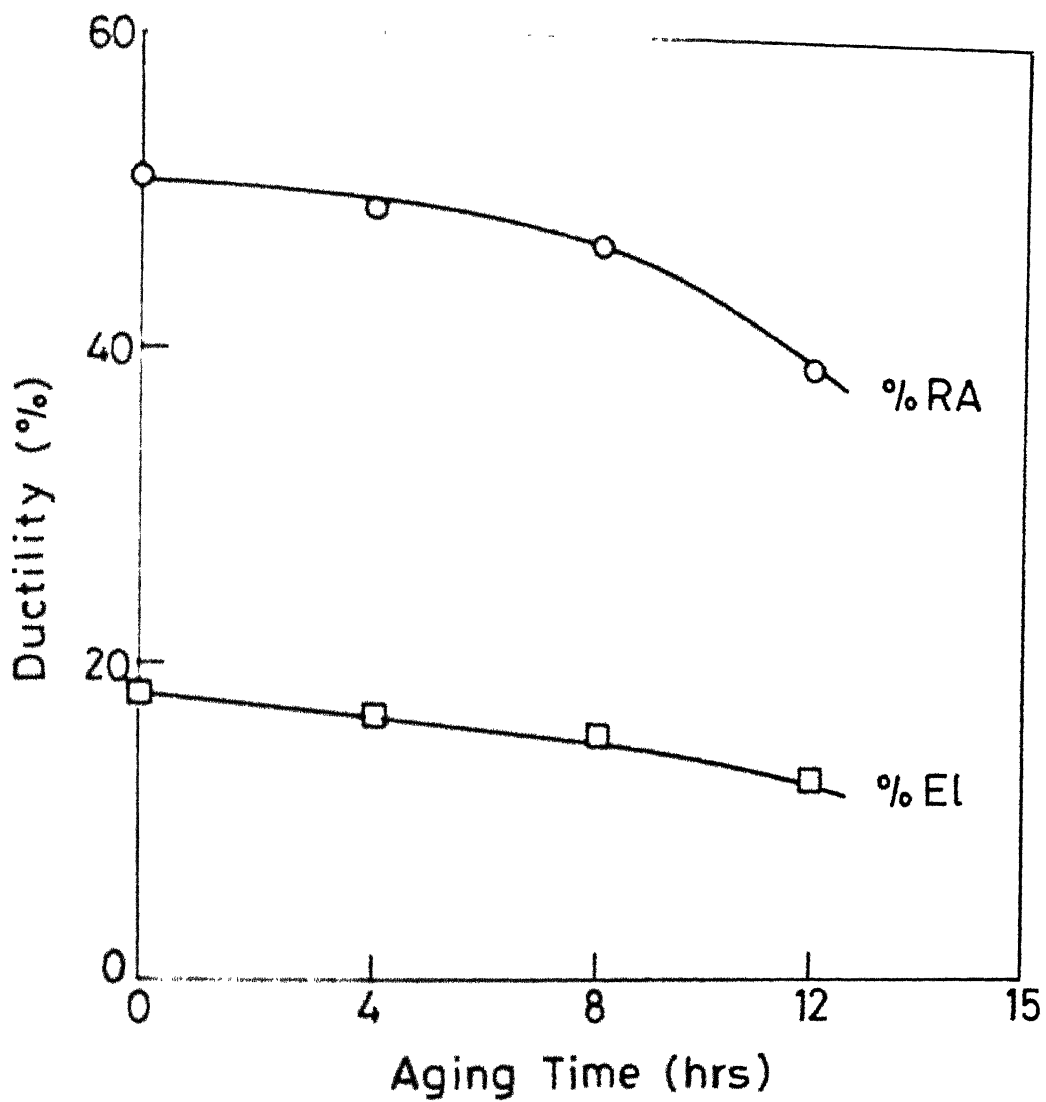


Fig.4.11 Effect of aging time on ductility aged at temperature 480°C.

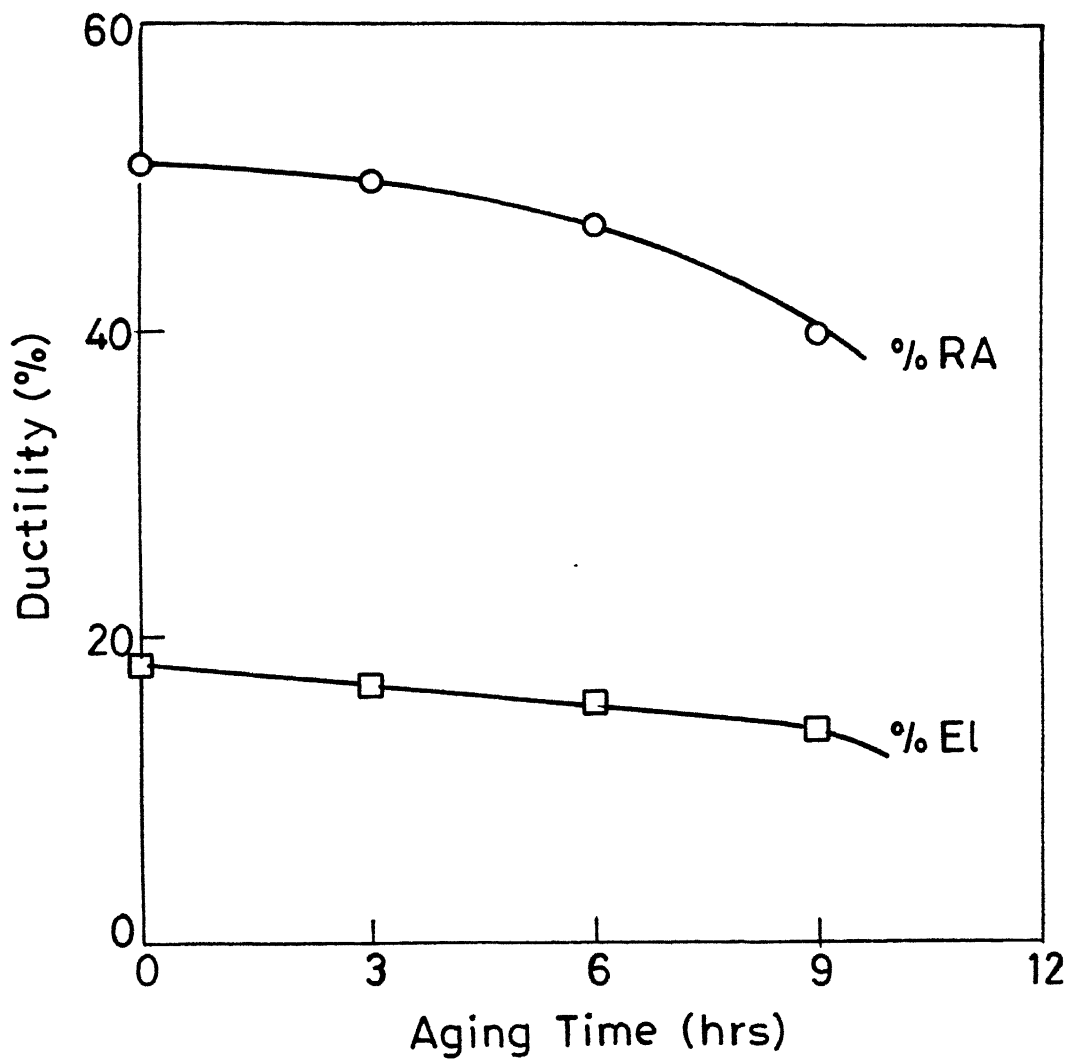


Fig.4.12 Effect of aging time on ductility aged at temperature 530 °C

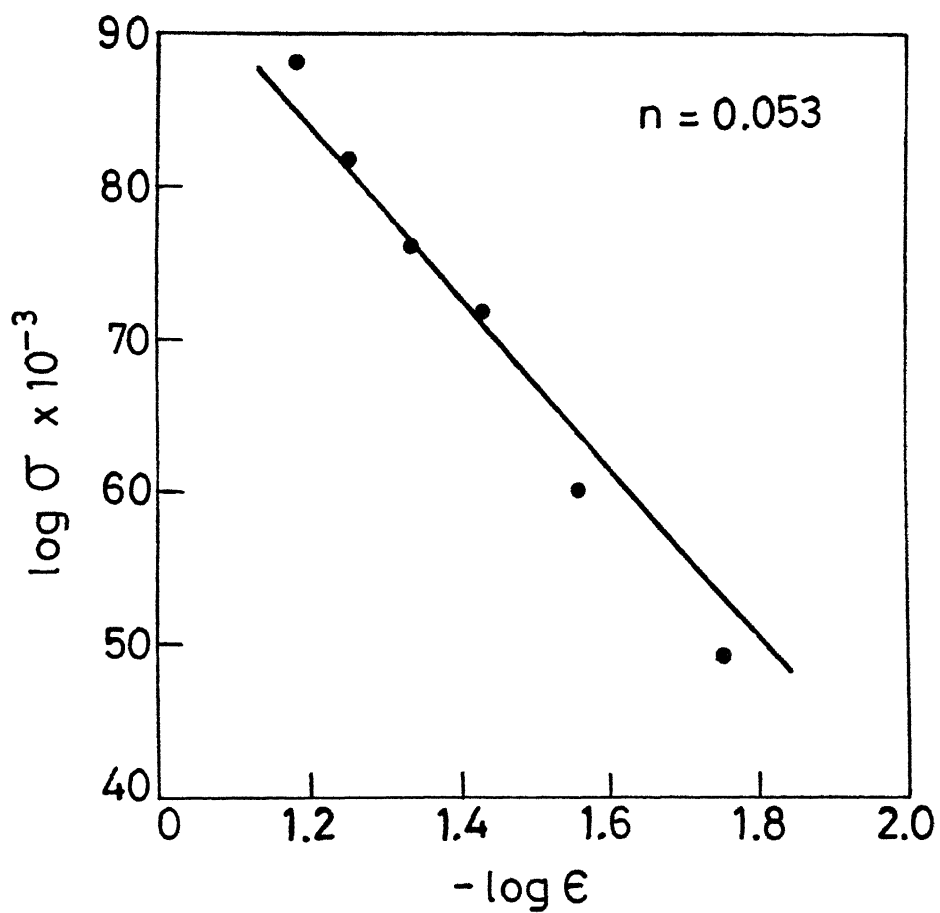


Fig.4.14 Log -Log plot of true stress -strain curve for a solution and aged specimen (950°C / 1hr / AC + 530°C / 6hr / AC)

Table 4.4 : Workhardening Parameter (n)

Thermal Treatment	Work Hardening Parameter
950 ⁰ C/1h/AC	0.041
950 ⁰ C/1h/AC+480 ⁰ C/4h/AC	0.050
950 ⁰ C/1h/AC+480 ⁰ C/8h/AC	0.053
950 ⁰ C/1h/AC+480 ⁰ C/12h/AC	0.055
950 ⁰ C/1h/AC+530 ⁰ C/3h/AC	0.052
950 ⁰ C/1h/AC+530 ⁰ C/6h/AC	0.053
950 ⁰ C/1h/AC+530 ⁰ C/9h/AC	0.054
950 ⁰ C/1h/AC+580 ⁰ C/1.5h/AC	0.051
950 ⁰ C/1h/AC+580 ⁰ C/4.5h/AC	0.053
950 ⁰ C/1h/AC+580 ⁰ C/7.5h/AC	0.055

parameters for the solution treated and solution treated plus aged samples. This increase may be attributed to the reduction in the mean free path of dislocations. However, the increase is not significantly large because of the void formation and crack propagation in the secondary α/β interface.

CHAPTER 5

CONCLUSIONS AND SUGGESTIONS FOR FURTHER WORK

5.1 Conclusions

1. The β transus temperature for the alloy BT-9 has been determined and found to be $\sim 1010^{\circ}\text{C}$.
2. Volume fraction of primary α changes with changing the solution treatment temperature and does not change significantly during aging treatment.
3. Grain size of primary α undergoes a small change does not change during aging while the precipitation of fine secondary α takes place during aging which is the main cause for improving the 0.2% PS, UTS and reducing the ductility.
4. The hardness does not change significantly with aging time, as no hard phase is precipitating during aging.
5. The aging treatment is to be given for stabilizing the microstructure for optimization of mechanical properties such as strength, ductility, fracture toughness, creep, fatigue etc. However, a more extensive study is necessary to study the effect of aging treatment for high temperature mechanical properties.

5.2 Suggestions for Further Work

1. TEM investigations and X-ray diffractions in detailed should be done to study the precipitation of secondary α morphology during aging.
2. Mechanical properties such as fracture toughness, stress

REFERENCES

1. J. Mathew and J.R. Donachie, Titanium and Titanium Alloys, Source Book, ASM, Ohio, 1982.
2. E.W. Coolings, Physical Metallurgy of Titanium Alloys, ASM, Ohio, 1984.
3. G.M. Meetham, Development of Gas Turbine Materials, 1981, pp.4-23 and 63-85.
4. S.M.L. Sastry et al., Titanium Science and Technology'80, Kyoto, Japan, p. 873.
5. Shakhanova et al., Titanium Science and Technology'80, Kyoto, Japan, p. 849.
6. K. Morii, H. Mecking and G. Luetjering, Proc. of Int. Conf. on Strength of Metals and Alloys, Pergamon Press, New York, 1985, p. 251.
7. I. Weiss, F.H. Froes, D. Eylon, G.E. Welch, Vol. 17A, Met. Trans. Nov. 1986, p. 1935.
8. Hirosuke Inagaki, Z. Metallkd, Aug. 1990, p. 540.
9. H. Margolin and Paul Cohen, Titanium Science and Technology'80, Kyoto, Japan, p. 1565.
10. M. Peters, G. Luetjering and Ziegler, Z. Metallka, May 1983, p. 282.
11. H.J. McQueen and J.J. Jonas: Treatise on Materials Science and Tech., Plastic Deformation of Materials, R.J. esenault ed. Academic Press, New York, Vol. 6, p. 393.
12. C.C. Chen and J.E. Coyne, Met. Trans. 1976, Vol. 78, p. 1931.
13. S.L. Semaitin and G.D. Lahote, Met. Trans. 1981, p. 1705.

14. J. Mathew and Donachie, Titanium, A Technical Guide, Jr. ASM, 1988.
15. J.C. Chesnutt, C.G. Rhodes and J.C. Williams, Fractography-Microscopy Cracking Processes, ASTM 1976, pp. 99-138.
16. J.C. Williams and E.A. Starke, Jr. Deformation, Processing and Structure, G. Krauss, ed. ASM, Metals Park, Ohio, pp. 279-354.
17. H. Margalin, J.C. William, J.C. Chesnutt and G. Luetjering, Titanium Science and Technology.
18. T.L. Trenogina and R.M. Lerinman, Proc. of 3rd International Conference on Titanium, Moscow, 1976, pp. 1623-1632.
19. J.C. Williams, Titanium Science and Technology, Plenum Press, New York, 1973, Vol. 3, p. 1433.
20. R.I. Jaffee, Titanium Science and Technology, Plenum press, New York, 1973, Vol. 3, p. 1665.
21. H. Kimura and O. Izumi, Titanium Science and Technology'80, Kyoto, Japan, 1980, Vol. 1, p. 163.
22. G. Welsch, G. Lutjering, K.G. Gazioglu and W. Bunk: Met. Trans. Jan. 1977, Vol. 8A, p. 169.
23. A.S. Ivanon and V.S. Tomsinsky, Phys. Met. Metall., 1973, Vol. 36, p. 102.
24. J.C. Williams, B.S. Hickman and D.H. Leslie, Met. Trans. 1971, Vol. 2, p. 477.
25. H. Margolin and P. Farrar, Ocean Eng., 1969, Vol. 1, p. 329.
26. H. Hamajima, G. Lutjering and S. Weissman, Met. Trans. 1973, Vol. 4, p. 847.
27. J.C. Williams and M.J. Blackburn, Trans. ASM, 1967, Vol. 60, p. 373.

28. Y. Ito, Private communication.
29. M.A. Greenfield and H. Margolin, Met. Trans., 1972, Vol. 3, p. 2649.
30. F. George Vander Voort, Metallography Principles and Practice, McGraw Hill, NY 1984.
31. R.T. DeHoff and F.N. Rhines, Quantitative Microscopy, McGraw Hill, NY 1968.
32. D. Banerjee and R.V. Krishnan, Proc. Indian Acad. Sci. (Engg. Sci.), 1988, Vol. 4, p. 21.
33. G.E. Dieter, Mechanical Metallurgy, Second ed., McGraw Hill, Tokyo, 1981, p. 340.

ME-1992-M-YAD-EFF